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DEVELOPMENT OF LOW TEMPERATURE GAS GENERATOR TECHNOLOGY

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Final Report

Dr. Donald R. Poole Rocket Research Corporation Seattle, Washington

December 1966

Air Force Rocket Propulsion Laboratory
Research and Technology Division
Air Force Systems Command
Edwards, California

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DEVELOPMENT OF LOW TEMPERATURE GAS GENERATOR TECHNOLOGY FINAL REPORT

Dr. Donald R. Poole

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FOREWORD

This technical report was prepared for the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards Air Force Base, California, by Rocket Research Corporation, Seattle, Washington, under Contract AF 04(611)-11376. This Final Report covers the period January 3, 1966, through November 14, 1966. The Air Force Project Officers were Capt. Joel A. Tolson and Lt. James R. Noyce. The Rocket Research Corporation Program Manager was Dr. Donald R. Poole.

This report has been assigned a secondary report number RRC-66-R-74 by Rocket Research Corporation.

This report contains no classified information extracted from classified documents.

This technical report has been reviewed and approved.

W. H. Ebelke, Colonel, USAF Chief, Propellant Division

ABSTRACT

The objective of this program was to characterize monopropellant hydrazine-based monopropellants which, by the use of ammonia and ammonia-water diluents, are capable of producing clean, low temperature gases when passed through a catalytic decomposition chamber. During the course of the p2-month program, thermochemical calculations were performed on a large number of cases involving various compositions of hydrazine, ammonia, and water. The effect of varying the amount of ammonia dissociation was investigated in the above calculations. Based upon the results of the thermochemical calculations and preliminary physical property testing, seven different solutions composed of various concentrations of hydrazine, water and/or ammonia were selected for further evaluation. The freezing points of the solutions were determined; and the vapor pressures, densities, and viscosities were measured over a wide temperature range. A low temperature gas generator was designed to produce approximately 60 standard cubic feet of gas per minute and to operate at a nominal chamber pressure of 300 psi. This gas generator was fired with each of the seven monopropellants in order to determine their steady state performance characteristics. In addition, a 1 lbf gas generator thruster was fired in pulse mode operation at various pulse widths and duty cycles with each of the seven monopropellants. The complete test results are presented in tabular form.

ARRPL-TR-66-226

TABLE OF CONTENTS

Section		Page
i	CONTRACT SCOPE	1
	1.1 General	3
	1.2 Thermochemical Calculations (Phase 1)]
	1.3 Propellant Characterization (Phase II)	1
	1.4 Gas Generator Testing (Phase III)	1
11	THERMOCHEMICAL CALCULATIONS	3
	2.1 Computer Program Description	- 3
	2.2 Chamber Conditions	3
	2.3 Thermochemical Data	4
	2.4 Discussion of Results	6
111	PROPELLANT CHARACTERIZATION	13
	3.1 Preliminary Evaluation	13
	3.2 Selection of Propellants	-18
	3.3 Physical Properties Determinations	18
	3.4 Summary of Propellant Physical Properties	33
IV	REACTOR TEST PHASE	41
	4.1 General	41
	4.2 Gas Generator Design	41
	4.3 Reactor Testing	. 45
	4.4 Results of Test Firings	52
	4.5 Summary and Conclusions	60
	APPENDIX	63
	REFERENCES	85

LIST OF FIGURES

Figure Number	•	?age
1	Heat of Solution, Ammonia-Hydrazine Solutions	7
2	Heat of Solution, Water-Hydrazine Solutions	8
3	Hydrazine-Water System, Chamber Temperature vs %H2O for Various Ammonia Dissociation	11
, 3a	Hydrazine-Water System, Chamber Temperature vs %H ₂ O for Various Ammonia Dissociation	12
Â.	Hydrazine-Ammonia System Chamber Temperature vs %NH ₃ for Varying Ammonia Dissociation	73
5	Hydrazine-Ammonia System Chamber Temperature vs %NH ₃ for Varying Ammonia Dissociation	74
. ś	Hydrazine, Equal Weight %H,O and NH, System Chamber Temperature vs % Additive for Varying Ammonia Disso- ciation	<i>7</i> 5
7	Hydrazine, Equal Weight %H ₂ O and NH ₃ System Chamber Temperature vs % Additive for Varing Ammonia Disso- ciation	76
8	Performance of the Hydrazine-Ammonia System as a Function of Temperature and NH ₃ Dissociation	77
. 9-	Performance of the Hydrazine-H ₂ O System as a Function of Temperature and NH ₃ Dissociation	<i>7</i> 8
10	Performance of the Hydrazine-Equal Wt %H ₂ O and NH ₃ System as a Function of Temperature and NH ₃ Disso- ciation	7 9
11.	70% Hydrazine, 30% NH ₃ System Reaction Product Composition	紉
12	50% Hydrazine, 50% NH ₃ System Reaction Product Composition	81
13	60% Hydrazine, 40% NH ₃ System, Reaction Product Composition	82
14	70% Hydrazine, 30% H ₂ O System, Reaction Product Composition	83
15	55% Hydrazine, 45% H ₂ O System, Reaction Product Composition	84
16	Freezing Point vs Weight % Additive for Various N ₂ H ₄ Solutions	15
17	Vapor Pressure vs Temperature 29.89% $\rm H_2O$, 30.07% $\rm NH_3$, 40.04% $\rm N_2H_4$	16

LIST OF FIGURES (Cont'd)

rigure Number		Page
18	Vapor Pressure vs Temperature, 60.06% H ₂ O, 9.87% NH ₃ , 30.07% N ₂ H ₄	17
19	Freezing Point vs Weight: H2O for N2H4 + H2O Solutions	22
20	Temperature Effect on Vapor Pressure of NoHa + H2O Solutions	23
21	Temperature Effect on Vapor Pressure of 40% N2H4/ 60% H2O	24
22	Temperature Effect on Vapor Pressure of 65.0% N ₂ H ₄ , 26.25% H ₂ C, 8.75% NH ₃	26
23	Temperature Effect on Vapor Pressure of Hydrazine-Ammonia- water Solutions	27
24	Rolling Ball Viscometer Schematic	28
25	Viscometer Loading Schematic	29
26	Temperature Effect an Viscosity of N2H4 + H2O Solutions	30
27	Temperature Effect on Viscosity of Hydrazine-Ammonia- Water Solutions	33
28	Temperature Effect on Viscosity of Hydrazine-Ammonia- Water Solutions	32
29	Temperature Effect on Density of NoHe + HoO Solutions	34
30	Temperature Effect on Density, 40.0% N.H., 60% H ₂ O and 65.0% N ₂ H ₄ , 26.25% H ₂ O, 8.75% NH ₃	35
31	Temperature Effect on Density, 45.0% N ₂ H ₄ , 27.5% H ₂ O, 27.5% NH ₃	36
32	Temperature Effect on Density, 35:0%.N ₂ H ₄ , 32.5% H ₂ O, 32.5% NH ₃	37
33	Temperature Effect on Density, 30.0% No. Ha, 70% NH3	.38
34	Low Temperature Gas Generator Assembly	42
35	LTGG Reactor Instrumentation	47
36	1 ibf Thruster Instrumentation	48
37	Reactor Test Schematic	49
38	Performance as a Function of Hydrazine Content	58
39	Characteristic Exhaust Velocity During Pulse Mode Operation	59

AFRPL-TR-66-226

LIST OF TABLES

Number		Page
1	Heat of Formation of Hydroxine Solutions	5
.	Theoretical Performance Hydrazine-Water System, Chamber Pressure = 380 psia, Exhaust Pressure = 14.7 psia	64
413-	Theoretical Performance, Hydrazine-Ammonia System, Chamber Pressure = 300 psia, Exhaust Pressure = 14.7 psia	67
榖	Theoretical Performance, 'Eydrazine-Equal Weight Percent' Water-Ammonia System	70
¥	Theoretical Performance Selected Hydrazine-Ammonia-Water Systems, Chamber Pressure = 300 psia, Exhaust Pressure = 14.7 psia	۶.
Al	Composition and Freezing Points of Hydrazine-Ammonia-Water Solutions	14
All	Compositions of Selected Low Temperature Gas Generator Propellants	19
ViII	Physical Properties of Low Temperature Gas Generator Propellants	.39
iX	Gas Generator Design Forameters	46
Х	Instrumentation Parameters	50
XI	Pulse Mode Firing Sequence	51
XII	LTGG Reactor Test Data	<i>5</i> 3
XIII	1 1bf Thruster Test Data Summary	54
XIA	Results of Exhaust Gas Analysis	61

SECTION I

CONTRACT SCOPE

1.1 General

This program was conducted in three phases. Phase I involved conducting themochemical calculations which provided theoretical data to assist in the selection of propellant systems for experimental evaluation. Phase II consisted of the determination of the physical properties of the selected propellants. Experimental delivered performance data was determined in Phase III.

This program was basically a propellant study and did not involve development of gas generator hardware, although a heavyweight experimental reactor was designed and fabricated for propellant evaluation purposes.

1.2 Thermochemis al Calculations (Phase I)

Thermochemical calculations were performed on various compositions for the hydrozine-ammonia-water system. The effect of varying amounts of ammonia dissociation was investigated in addition to the effect of varying the chamber pressure.

1.3 Propellant Characterization (Phase III)

Seven different solutions composed of various concentrations of hydrazine, water and/or ammonia were selected for characterization studies. The selection of these propellants was based on the results of the thermochemical calculations and an preliminary physical property testing. The freezing point, density, vapor pressure and viscosity of each propellant was then measured.

1.4 Gas Generator Testing (Phase III)

A heavyweight experimental reactor was designed and fabricated. The reactor operated at a nominal chamber pressure of 250 psia and produced approximately 60 standard cubic feet of gas per minute. Each of the seven propellants was evaluated using this reactor.

SECTION II

THERMOCHEMICAL CALCULATIONS

2.1 Computer Program Description

The computer facilities of the Service Bureau Corporation, Palo Alto, California, were utilized for Phase I thermochemical equilibrium analyses. The NASA computational procedure was used for solution of the basic problem, as published in NASA Report No. 1037. The thermochemical data employed in this analysis was obtained from the joint Army, Navy, Air Force (JANAF) Interim Thermochemical Tables.

2.2 Chamber Conditions

The decomposition of hydrazine may be considered to take place according to the following consecutive reactions:

$$3N_2H_4 \rightarrow 4NH_3 + N_2 \tag{1}$$

$$2NH_3 \longrightarrow N_2 + 3H_2 \tag{2}$$

For the purpose of calculating the relative amounts of the reactants, it was assumed that equation (1) goes to completion. The extent to which reaction (2) takes place is determined by the efficiency of the catalyst used and the design details of the reaction chamber.

Reactions (1) and (2) may be combined as follows:

$$N_2H_4 \rightarrow 4/3 (1-X) NH_3 + \left(\frac{2X+1}{3}\right) N_2 + 2XH_2$$
 (3)

Where

 $X = fraction of NH_3 dissociated.$

For the cases in which various concentrations of ammonia in hydrazine were considered. X represents only the fraction dissociation of the ammonia which was derived from the decomposing hydrazine, and Y represents the fraction of the added ammonia which has dissociated.

The input compositions were calculated in units of gram-atoms per 100 grams of reactant for hydrogen, oxygen, and nitrogen and gram-moles per 100 grams of reactant for ammonia by means of the following equations:

$$\frac{4(1-X)}{3} \left(\frac{P_h}{32.04E} \right) + (1-Y) \left(\frac{P_{cm}}{17.032} \right) = (NH_3)$$
 (4)

$$\frac{2(1-X)}{3} \left(\frac{P_h}{32.048}\right) + Y \left(\frac{P_{am}}{17.032}\right) = (N)$$
 (5)

$$\frac{2P_{w}}{18.016} + \frac{4P_{h}X}{32.048} + \frac{3P_{cm}Y}{17.032} = (H)$$
 (6)

$$\frac{P_{w}}{18.016} = (0) \tag{7}$$

Where:

Ph = weight percent hydrazino in reactants

P = weight percent ammonia in reactants

P. - weight percent water in reactants

X = fraction ammonia (derived from hydrazina) dissociated

Y = fraction of added ammonia dissociated

Calculations were performed for numerous propositions compositions where arbitrary amounts of ammonia were impressed upon the reaction products in the chamber. In these cases, ammonia was not allowed to dissociate and the formation of additional ammonia was not permitted. For practical purposes this condition specified the composition of the chamber gases for these reactants. Numerous calculations were also performed in which complete chemical equilibrium was allowed to exist. Equilibrium conditions were applied whenever X = Y = 1.

All calculations were carried out under frozen flow conditions. Calculations under shifting flow conditions were not necessary since the results would be identical to frozen flow conditions for all practical purposes.

2.3 Thermochemical Desa

The heats of formation used in the thermochemical calculations are listed in Table 1. Thes values were derived from the standard heat at formation of each component of the solutions and the heats of solution of ammonia and water in hydrazine.

TABLE I
HEAT OF FORMATION OF HYDRAZINE SOLUTIONS

Weight % N ₂ H ₄	Weight % NH ₃	Weight % H ₂ O	Δ H kcais/100 gm
100	O	0	+ 37.5998
90	Q	10	- 5.5018
70	0	30	- 90.7060
65	0	35	-111.4167
60	0	40	-132.9876
55	0	45	-153.5183
50	0	50	-174.4393
45	О,	55	-195.3601
40	0	60	-216.2509
30	0	70	-258.0026 c
95	5	0	+ 30.399
90	10	0	+ 24.508
80	20	0	+ 11.486
70	30	0	- 1 <i>.55</i> 2
60	40	0	- 14.558
55	45	0	- 21.069 '
50	50	o	- 2 7.580
40	- 60	0	- 49.602
35	65	0	- 47.113
30	70	0	- 53.624
80	10 -	10	- 18.691

Values of the heat of solution for various concentrations of ammonia in hydrazine were obtained from the data given in Reference 3. After conversion to the appropriate whits, this information was plotted as shown in Figure 1. This graph indicates that the heat of solution is essentially constant for concentrations greater than about 5%. The value -0.07 kcal/100 g was therefore applied as a correction in all cases involving ammonia solutions, even though a correction of this magnitude has very little influence on the results:

Corrections for the heat of solution for the various concentrations of water in hydrazing were obtained from Figure 2 (References 2 and 3).

For those solutions containing both ammonia and water in hydrazine, the heats of solution were estimated from values for the two component systems. The constant value of C.07 kcgl/100 g was applied to correct for the heat of solution of ammonia in hydrazine. The concentration of water in hydrazine was calculated as though no ammonia was in the solution and the corresponding heat of solution was then obtained from Figure 2.

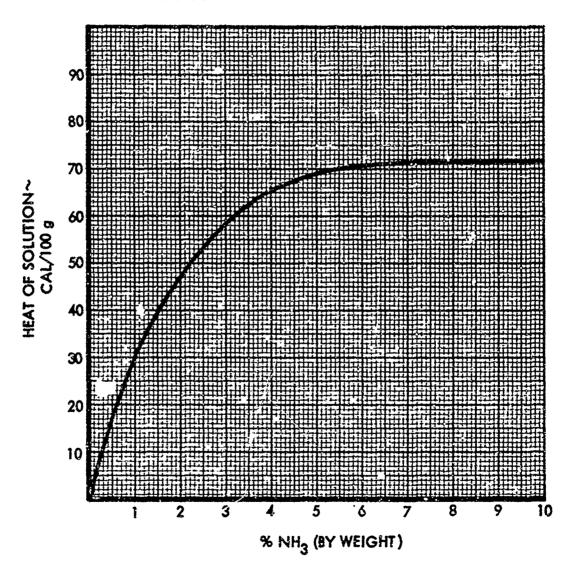
Thermochemical calculations were perfermed on a test case in an effort to determine the significance of the heat of solution corrections. The test case consisted of 30% ammonia and 70% hydrazine with X = .4 and Y = 0 and was calculated both with and without the heat of solution correction. When the correction was applied, the chamber temperature was 931.08°K and the characteristic exhaust velocity was 3,559 ft/sec. Without the correction, these values were 932.11°K and 3,561 ft/sec. From these values it was concluded that any errors which may have been introduced by the described methods of correcting for the heat of solution would not influence the results significantly.

2.4 Discussion of Results

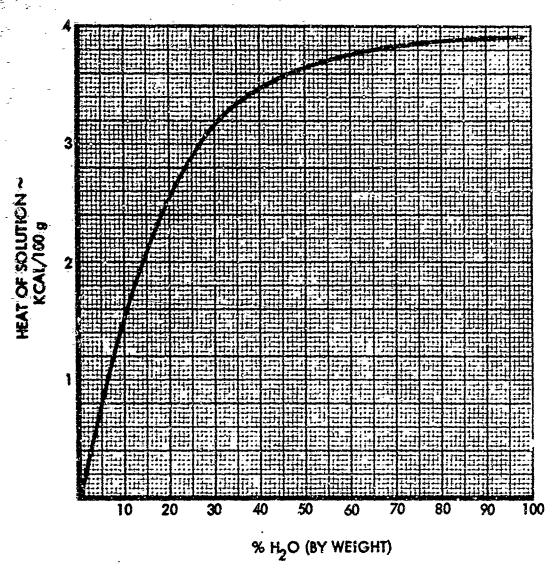
The results of the Phase I thermochemical calculations are summarized in Tables II, III, and IV and in Figures 4 through 15 respectively, in the Appendix. These figures illustrate the wide ranges of chamber temperatures and performance which are theoretically possible through the use of mixtures of ammonia, water, and hydrazine.

During a later phase of the program, after the selection of the propellant compositions which were characterized and tested in the reactor, another series of calculations was performed. These calculations, summarized in Table V, provide a more direct comparison with the results of the reactor testing (Phase III) than was possible with the

HEAT OF SOLUTION AMMONIA - HYDRAZINE SOLUTIONS



HEAT OF SOLUTION WATER - HYDRAZINE SOLUTIONS



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TABLE V
YHEORETICAL PERFORMANCE
SELECTED HYDRAZINE-AMMONIA-WATER SYSTEMS
CHAMBER PRESSURE = 300 psis = EXHAUST PRESSURE = 14.7 psis

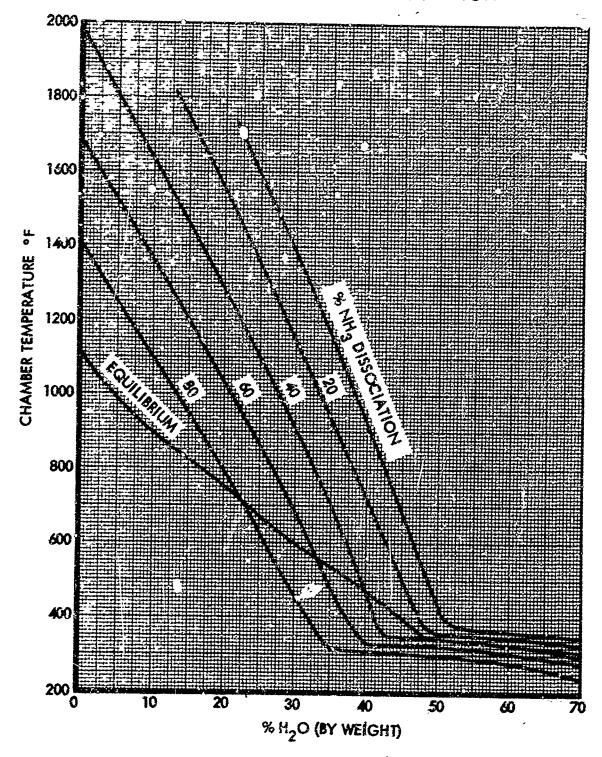
	,							~								A
Moles H ₂ O (Liquid)	:	:	1	:	1.413	1,776	2.128	:	:	:	;	;	:	;	0,1001	0,8787
Moles H ₂ O (Ges)	1,7040	1.7040	1,7040	2.7198	1.918	1.554	1.202	;	:	;	1,4570	1,4570	1.4570	1.3264	1.4363	0.9252
Moles NH ₃	2.3832	2,3085	2.72%	2,1218	1.654	1.33)	966,0	5,358	5,108	4.859	3.2180	2,6771	2.1363	3,4858	3.1121	3,3643
Moles N2	0.7208	0.9832	1.2456	0.5305	0.416	0.582	0.749	0.312	0.437	0.562	0.6761	0,9465	1,2169	0,4681	2,3402	0.3640
Moles 7	;	0,8650	2.7300	:	;	0.459	0.999	1	0,374	0,749	1	0.8134	1.6228	;	0,5617	:
Frozen Gerrana (Ch.)	1,2101	1,2463	1,2860	1.2940	1,154	1,139	1.127	1.282	1,283	1.320	1.2118	1.2472	1.2862	1,2823	1,3109	1,1945
₩.W.	18,839	16,945	15.373	18.615	18.482	17,411	16,438	17.636	16.893	16.209	18,680	16.972	15.545	18.244	19.522	18.076
AHR Keel/ 100gm	-24.469	-22.965	-20.782	-12.864	966'8 -	- 8,985	- 8.744	-10,122	- 9.033	- 7.892	-23,100	-21,467	-19.403	-12.828	-15,184	- 9.756
~8-X	146.45	141.88	135.46	105.80	7.	87.9	87.2	93.8	形.7	82.9	141.80	136.67	129.93	105.65	94.74	23.14
3#5/iJ	3364	3261	31.50	2384	288	1978	1957	27.22	2062	1922	3240	3162	3021	23%5	2132	200
الرساة	1362	1133	8	697	8	₹	SS	822	179	8	1240	1024	9 0	473	283	38
7°~~3	2101	883	78	225	35	447	436	410	જ	305	776	824	ğ	81%	418	<u>2</u> .
٨	;	1	ŀ	i	1	;	;	1	1.	1	:	;	;	í	ł	1
×	0.0	0.2	4.0	0.0	0.0	0.3	4.0	0.0	0.3	4.0	0.0	0.2	4.0	0.0	0.2	0.0
% H ₂ O	20.2	8.	30.7	\$	8	8	8	:	:	f	3.75	8.73	8,75	2.3	2 .3	32.5
% NH3	;	;	ł	ŧ	į	î	:	2	2	8	26.23	28.25	28.25	27.5	27.5	32.5
* N2H4 % NH3 % H2O	66.3	69.3	69.3	જ	ş	\$	\$	8	8	8	3	3	3	\$	£	×
Code No.	3.307.00	3.307.20	3.307.40	3.49.00	3,40.00	3.60.20	3,60,40	4.70.00	4,70.20	4.70.40	7.2625.675.00	7.2623.875.20	7.2623.875.40	7.275.275.00	7.275.275.20	7,325.325.00
Kcol/ 1009	-93.520	-93.520	-73.530	120.281	-216.251	-216.251	-216.331	-53.624	-53.624	-53.624	-36.346	-86.346	-86.546	-116.275	-116,293	-143.661

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previous calculations. It is interesting to note the abrupt changes in the slopes of the curves in Figures 3 and 3a. These changes are assumed to be due to the (theoretical) condensation of water in the chamber.

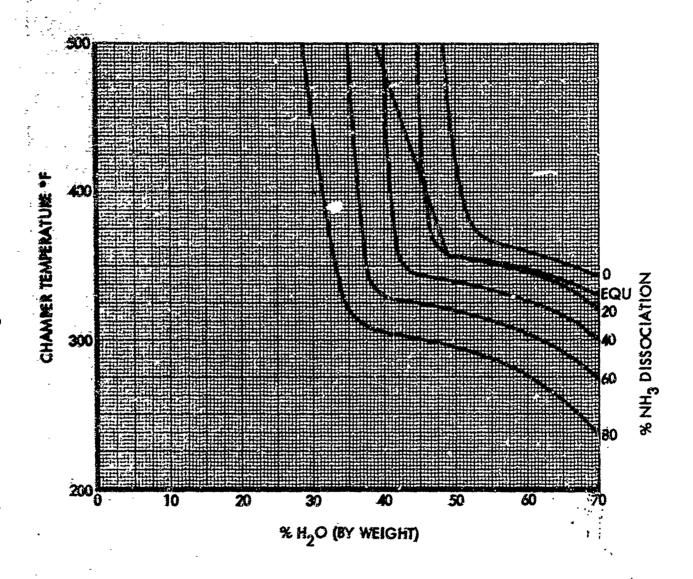
HYDRAZINE - WATER SYSTEM

CHAMBE TEMPERATURE VS % H2O FOR VARIOUS AMMONIA DISSOCIATION



HYDRAZINE - WATER SYSTEM

CHAMBER TEMPERATURE VS % H₂O FOR VARIOUS AMMONIA DISSOCIATION



SECTION III

PROPELLANT CHARACTERIZATION

3.1 Preliminary Evaluation

Preliminary testing was conducted to generate information required in the selection of solutions for further study.

Solutions with various concentrations of hydrazine, ammonia and water were sealed in heavy-wall, glass tubing and the freezing points of the solutions were determined. The compositions and freezing points of these solutions are listed in Table VI. Figure 16 illustrates this information and includes the freezing point curve for the hydrazine-water system taken from the work of Hill and Sumner (Reference 4).

It is interesting to note that solutions having a water to ammonia ratio (by weight) larger than one have lower freezing points than solutions containing only water and hydrozine at the same percent additive. Solution GT-5 would not freeze at -110°F although it was packed in dry ice overnight. This solution was also cooled in liquid nitrogen, but a glass-like material resulted and no freezing point was observed.

Although it was considered unlikely that any phase separation would occur, the samples numbered GT-1 through 5, 8, and 11, which were used for the freezing point determinations, were placed in individual lucite tubes and then placed in a temperature-controlled oven. The door of the oven was replaced by a lucite plate, and the temperature was increased in small increments up to 160°F. Each temperature was held constant for at least one-half hour, and the samples were held at 160°F overnight. A light was placed behind the samples to facilitate viewing. The samples were observed viscally, and no evidence (such as opalescence or a second liquid layer) of the appearance of a second liquid phase was observed.

It may be concluded, therefore, that hydrazine, ammonia, and water are completely miscible at temperatures from the freezing point up to 160°F and over the concentration range of interest for this study.

The vapor pressure of two hydrazine, water, and ammonia solutions was measured at various temperatures. This information is illustrated in Figures 17 and 18.

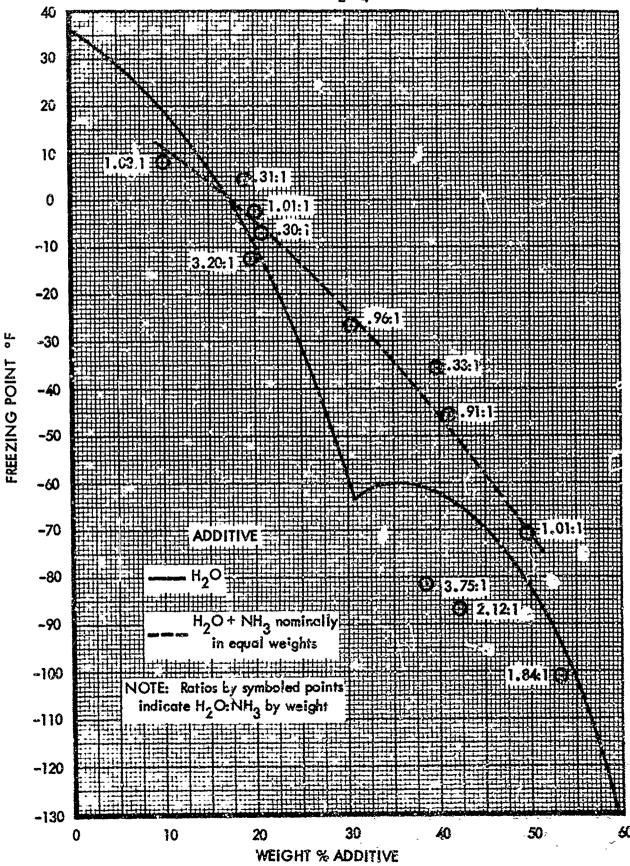
The methods used for these measurements is described in Paragraph 3.3.

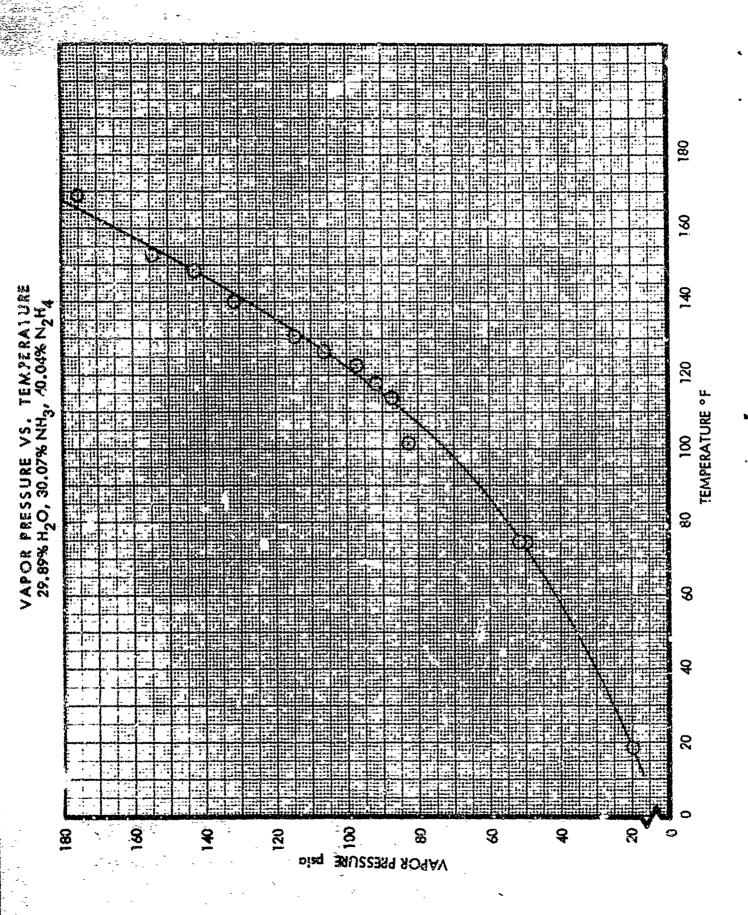
TABLE VI

COMPOSITION AND FREEZING POINTS OF
HYDRAZINE-AMMONIA-WATER SOLUTIONS

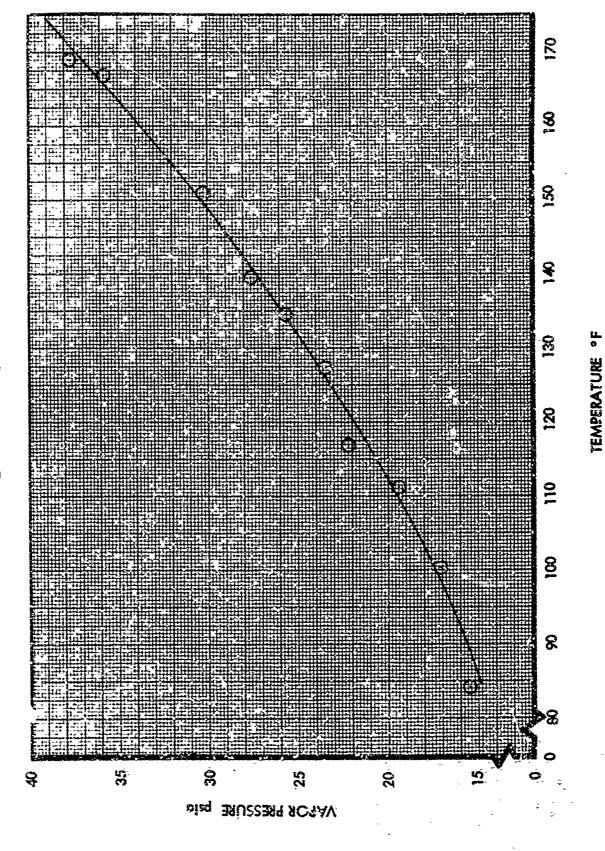
Solution		Freezing		
Designation	H ₂ O	NH ₃	N ₂ H ₄	Point °F
GT-I	4.51	14.34	81.15	+4
GT-2	30.50	8.14	61.36	-82
GT-3	19.62	21.50	58.87	-46
GT-4	14.92	15.47	69.61	-27
GT-5	30.55	28.71	40.74	<- 110
GT-6	5,03	4.87	90.10	⊹ 8
GT-7	10.01	9.92	80.07	-3
G7-8	25.08	24.76	50.16	,71
GT-9	34.70	18.82	46.48	-101
GT-10	28.71	13.53	57.76	87
GT-11	4.78	15.97	79.25	-7
GT-12	14.89	4.66	80.45	-13
GT-13	9.88	29.96	69.16	-36

FREEZING POINT VS WEIGHT % ADDITIVE FOR VARIOUS N2H4 SOLUTIONS





VAPOR PRESSURE VS TEMPERATURE 60.06% H₂O, 9.87% NH₂, 30.07% N₂H₄



22038

- 17 -

FIGURE 18

3.2 Selection of Propellants

After consideration of the thermochemical calculations and the information described in Paragraph 3.1, seven low temperature gas generator propellants were selected for further evaluation. The physical properties of these propellants were measured as a function of temperature, and their performance was determined in reactor firings.

The compositions of the solutions were chosen in order to provide estimated chamber temporatures over the range of 275°F to 925°F, to have low freezing points, and to provide data on a variety of propellant compositions. The compositions, estimated chamber temperatures, and measured freezing points are listed in Table VII.

3.3 Physical Properties Determinations

3.3.1 Preparation of Propellants

Anhydrous hydrazine obtained from Olin Chemicals was analyzed by the J.M. Kniseley Engineering Company, Seattle, Washington, and the results of the analysis were utilized in calculating the concentration of water in the solutions which were mixed.

Ammonia, obtained from the J. T. Baker Chemical Company and specified as 99.99% ammonia, was used as received.

For the preparation of solutions to be used for freezing point determination, glass tubes approximately 10 inches long were prepared from tubing having an inside diameter of 8 mm and a wall thickness of 1.5 mm. The tubes were sealed on one end and a constriction was placed approximately two inches from the open end by heating the tube until the walls softened and collapsed, leaving a passageway of approximately 2 mm.

After weighing the empty glass tube on an analytical balance, the appropriate amount of hydrazine-water solution was placed in the lower part of the tube by means of a micropipet and the tube was then reweighed. The tube was then attached to the vacuum manifold by means of a short rubber vacuum tube. The hydrazine water solution was frozen by immersing the tube in liquid nitrogen and the tube was evacuated. Ammonia was then metered into the vacuum manifold to slightly less than atmospheric pressure as indicated by a mercury manometer. The vacuum manifold has two, one-liter flasks attached by stopcocks so that either one, or both, flasks

TABLE VII

COMPOSITIONS OF SELECTED LOW TEMPERATURE
GAS GENERATOR PROPELLANTS

Solution	Composi	tion % by	Weight	Estimated	Measured		
No.	N ₂ H ₄	H ₂ O	Chamber Temp.		Freezing Point		
LT-1	69.3	30. <i>7</i>	ನೇಳು	925	-64		
LT-2	45	27.5	27.5	500	-80		
LT-3	35	32.5	32.5	275	-104		
LT-4	40	60	*****	350	-130		
LT-5	51	49		500	-80		
LT-6	30		70	300	-75		
LT-7	65	26.25	8.75	900	-57		

can be included in the volume of the manifold. The manifold volume was calibrated by absorbing ammonia in water and noting the increase in weight of the solution and the decrease in pressure within the manifold. The pressure change required to deliver the desired amount of ammonia was calculated, and the ammonia was then condensed into the glass tube by cooling with liquid nitrogen. The tube was then socied off, and both parts were weighed after the tube warmed to room temperature. The actual amount of ammonia in the solution was, therefore, determined by weight.

Solutions which were mixed in larger quantities for vapor pressure, viscosity, and density measurements were prepared in stainless steel cylinders. The hydrazine—water solution was drawn into an evacuated, weighed cylinder which was then re—weighed to determine the exact amount of solution. Lightly more than the calculated amount of ammonia required for the solution was then condensed into a separate cylinder. The exact weight of ammonia was then adjusted, after warming to room temperature, by alternately bleeding off ammonia and weighing the cylinder. The weighings were performed on a Mettler precision top-loading scale capable of weighing to ± 0.02 gm.

The hydrazine—water solution was then frozen in liquid nitrogen and the ammonia was condensed into the solution cylinder. After warming to room temperature, the solution was mixed by vigorous shaking.

3.3.2 Freezing Point Determinations

The freezing points of the solutions containing ammonia were determined by the technique described below.

An accetone cold-bath was prepared in a large, clear-glass Dewar flask. A spark-proof stirrer provided the agitation necessary to keep an even temperature throughout the bath. The sample tube was handled by means of a small apparatus clamp with vinyl fingers. The sample was partly frozen by immersing in a separate dry ice bath and then was completely immersed in the large cold-bath. The tube was then rocked back and forth by hand and the crystals were viewed, with the aid of a strong light, through the walls of the clear Dewar flask. If the crystals melted, the bath was cooled by adding dry ice and the process was repeated. If the solution continued to freeze, the bath was warmed slightly by a small immersion heater. The freezing point was thus bracketed, and the process was repeated until the freezing point was determined as accurately as required. The freezing point is defined as the temperature at which

the last solid disappears upon slowly warming the solution. Due to the large volume of the acetone bath (approximately four gallons) the temperature changes very slowly when adding dry ice or heating and it is possible to control the temperature easily to a small range. The accuracy of freezing points determined by this method is believed to be about $\pm 0.5^{\circ}$ F for temperatures down to about $\pm 60^{\circ}$ F and about $\pm 1.0^{\circ}$ F for lower temperatures. All thermometers used in these tests were certified to conform to ASTM Standards.

The solutions containing 27.5 and 32.5 per cent ammonia exhibited a marked tendency to supercool and considerable difficulty was experienced in freezing the solutions. Crystallization of both solutions was accomplished, however, and the freezing points were obtained by observing the disappearance of the last crystals upon slowly warming the solutions.

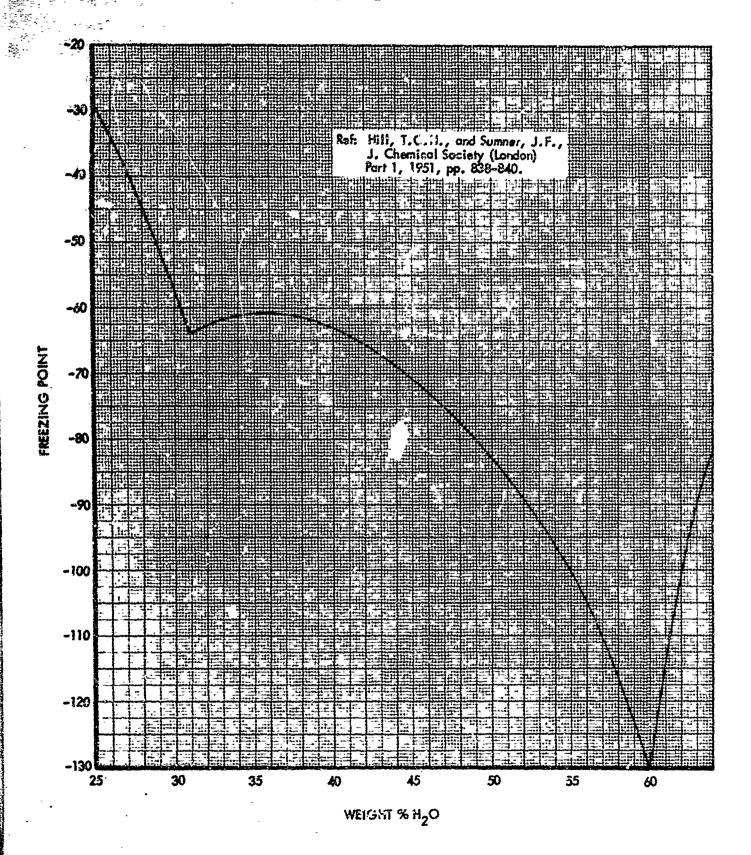
Freezing points of the solutions containing only hydrazine and water were taken from the data of Hill and Sumner (Reference 4). A part of their data was plotted on an expanded scale as shown in Figure 19, and the freezing points were taken from this graph.

3.3.3 Vapor Pressure Measurements

The vapor pressures of the solutions containing only hydrazine and water were measured in an all-glass apparatus consisting of a glass bulb, which holds the solution, connected to a short mercury manometer. The mercury was degassed by heating with a hand torch while evacuating the system. The solution was then added to the bulb and degassed by a freeze-evacuate-thaw-procedure which was repeated three times; the apparatus was then sealed while being evacuated. The complete apparatus was then immersed in a controlled temperature bath, and the mercury level was read by means of a cathetometer. The results of these measurements are shown in Figures 20 and 21.

The vapor pressures of the solutions containing ammonia were measured by means of a 0-300 psi, temperature compensated, Heise pressure gauge. The stainless steel cylinder containing the solution was connected to the pressure gauge through a 0.25 inch stainless tube and tee. The other end of the tee was closed by a small valve through which the apparatus, except for the cylinder, was evacuated. For temperatures below ambient, the cylinder was immersed in a cold bath and, at

FREEZING POINT VS WEIGHT % H_2O FOR $N_2H_4 + H_2O$ SOLUTIONS

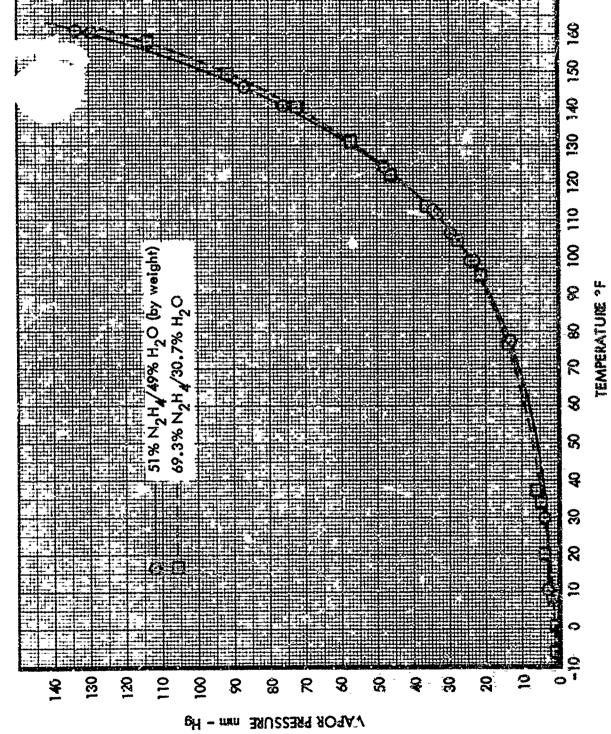


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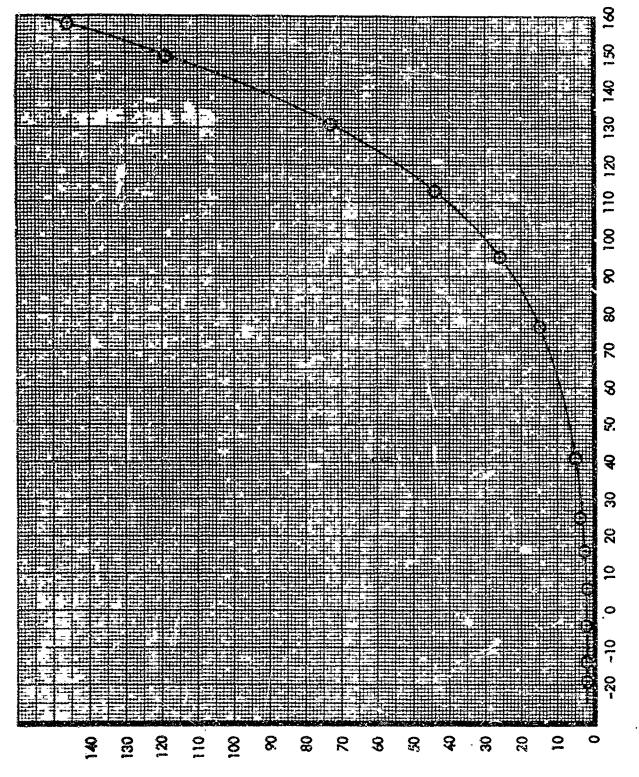
- 22 -

FIGURE 19

TEMPERATURE EFFECT ON VAPOR PRESSURE OF N2H4 + H2O SOLUTIONS



OF 40% N2H4/60% H2O



VAPOR PRESSURE mm -

FIGURE 21

each temperature, was shaken until the pressure remained constant. For temperatures above ambient, the cylinder and pressure gauge were placed in a temperature controlled oven which is capable of maintaining the temperature constant to within ± 1°C. The temperature was increased in increments and was held constant at each temperature until the pressure remained constant for at least 15 minutes. The vapor pressures of the solutions containing ammonia are illustrated in Figures 22 and 23.

3.3.4 Viscosity Measurements

Viscosity measurements were performed on solutions containing only hydrazine and water by using container-Fenske glass capillary viscometers. The measurements were conducted according to ASTM Standard Test Method D445-64-IP71. The viscometers were calibrated by the Cannon Instrument Company, State College, Pennsylvania.

The above method is not adequate for measuring the viscosity of solutions containing ammonia because of the higher vapor pressures which necessitated devising a new technique for these measurements. The apparatus, shown schematically in Figure 24, is an adaptation of the conventional rolling-ball viscometer. The viscometer consists, essentially, of a heavy-wall glass capillary tube with a small steel sphere which rolls down the inclined tube. The sphere is pulled to the top of the tube by means of a small magnet and then released. The time of travel of the sphere between two marks on the tube is measured by a stopwatch. The tube is held at a reproducible angle by a special clamp. This viscometer was calibrated with distilled water and a standard oil obtained from the Cannon Instrument Company.

The viscometer is loaded by a gravity flow arrangement shown schematically in Figure 25. The viscosities of the various solutions are illustrated in Figures 26, 27, and 28.

3.3.5 Density Measurements

Density measurements were run on the solutions containing only hydrazine and water by using the Lipkin Bicapillary pycnometer as specified in ASTM Standard Test Method D941-55. This method is not adequate for the solutions containing ammonia due to the higher vapor pressures and another technique was used.

A new pycnometer was fabricated which is similar to the Lipkin Bicapillary pycnometer except that heavy-wall glass capillary tubing was used and the ends of

TEMFERATURE EFFECT ON VAPOR PRESSURE OF 65.0% N₂H₄, 26.25% H₂O, 8.75% NH₃

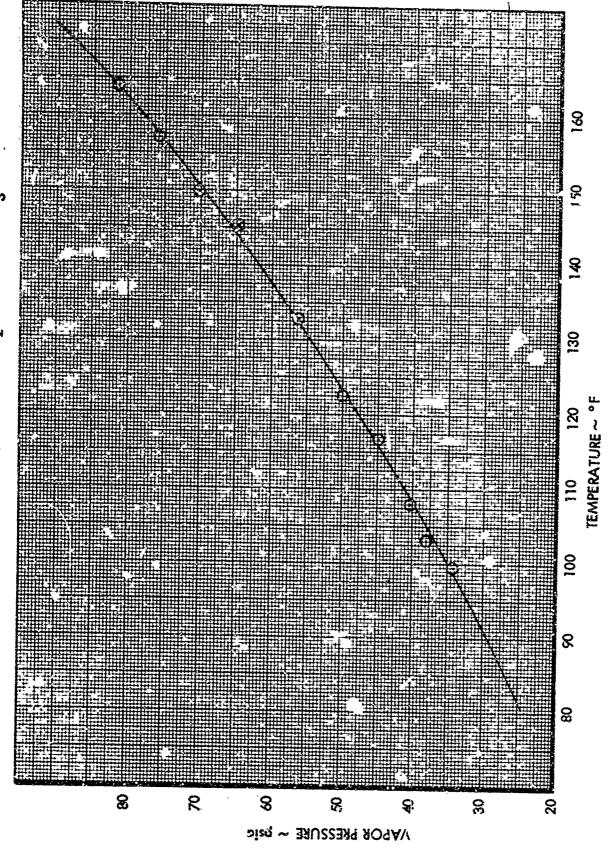
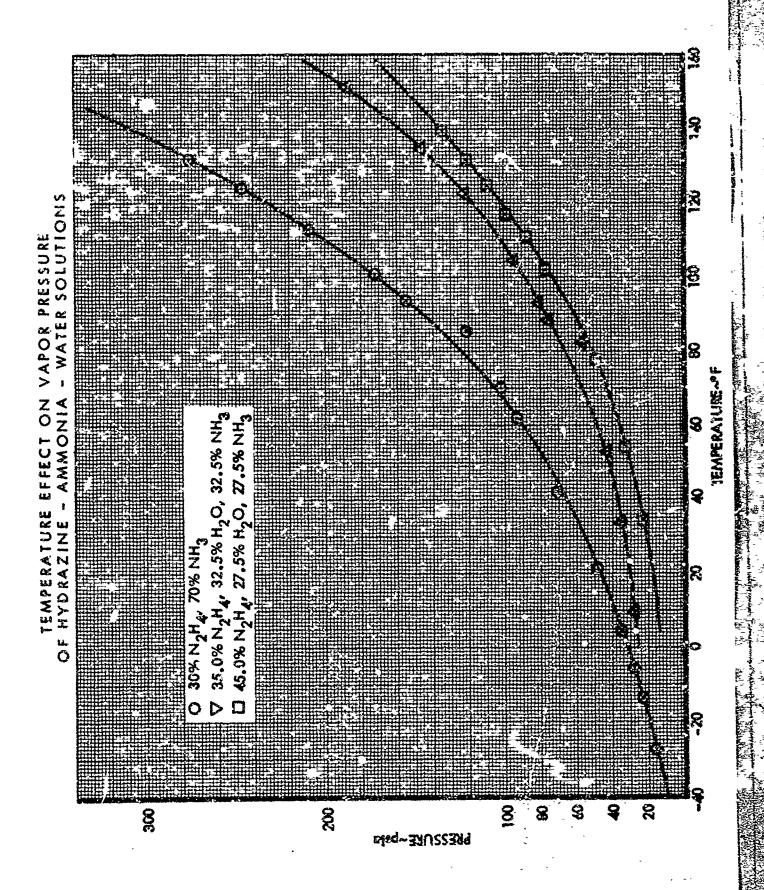


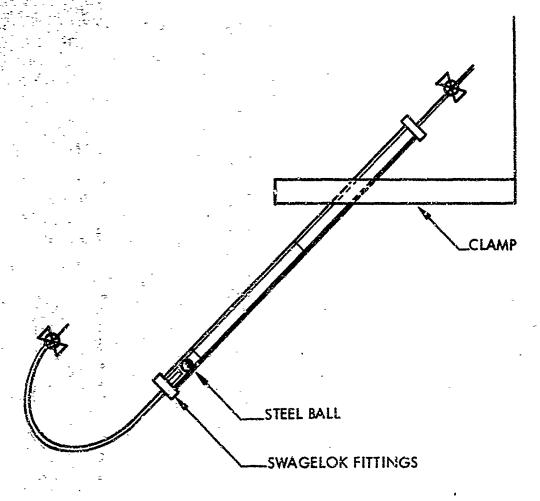
FIGURE 22

- 26 -

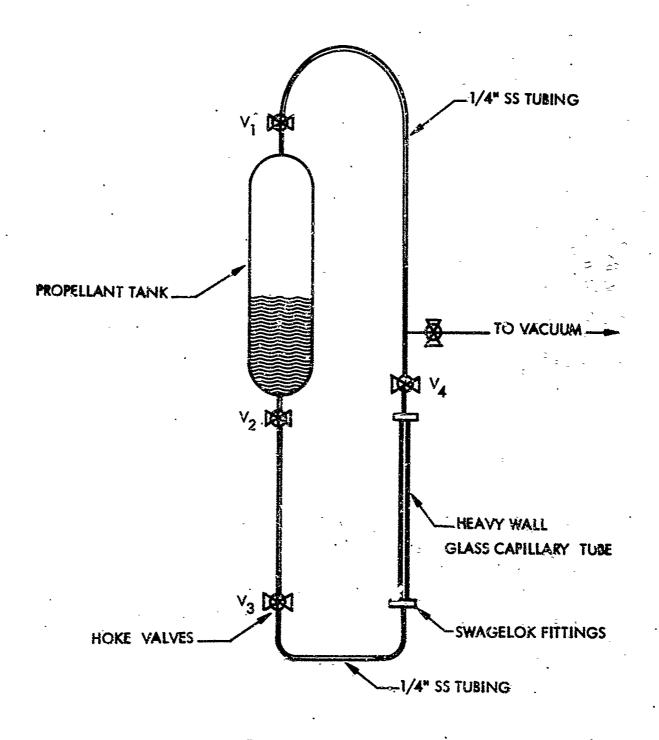
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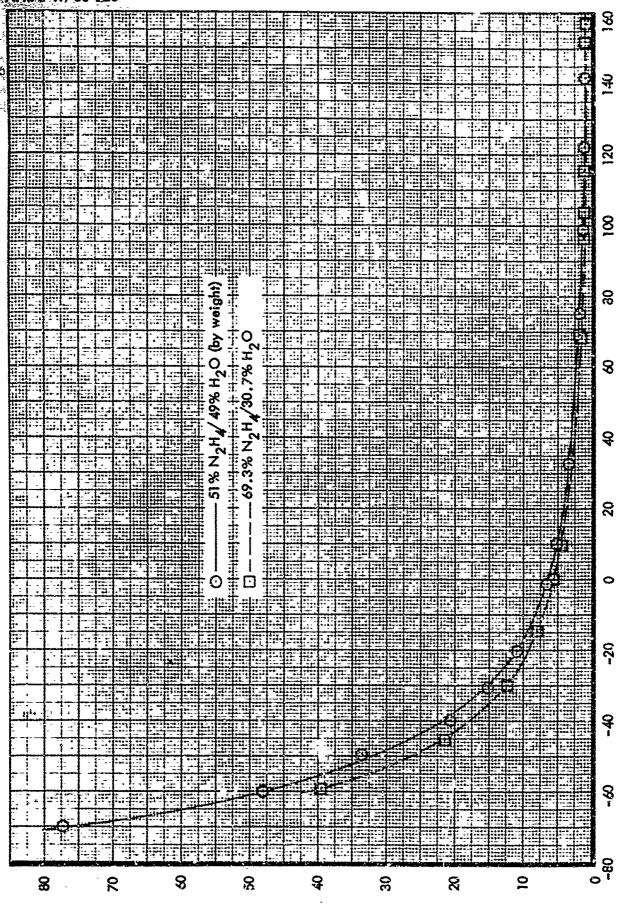


ROLLING BALL VISCOMETER



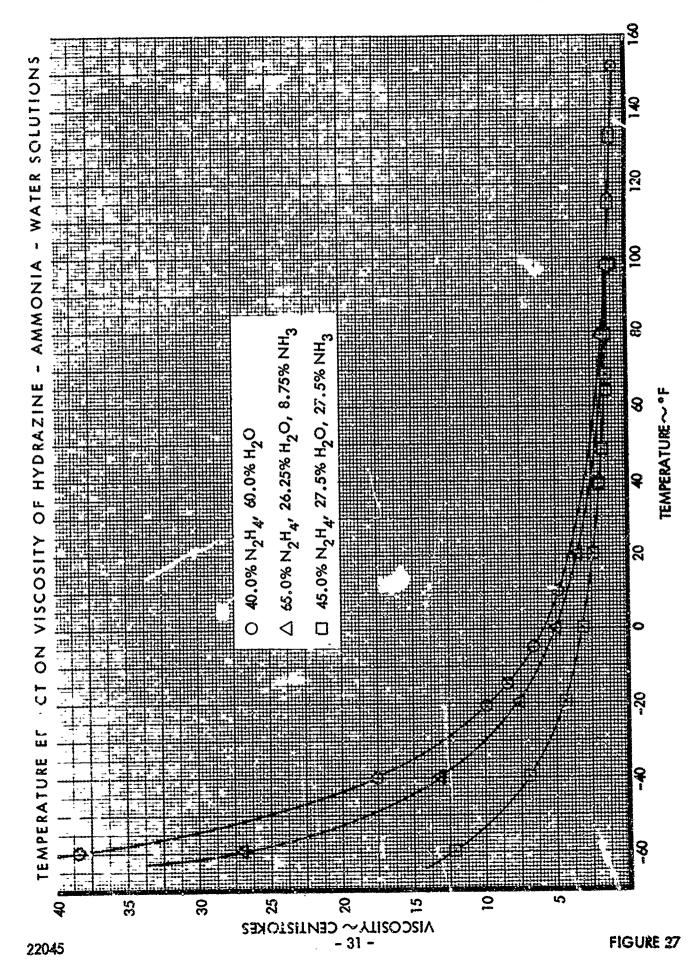
VISCOMETER LOADING SCHEMATIC



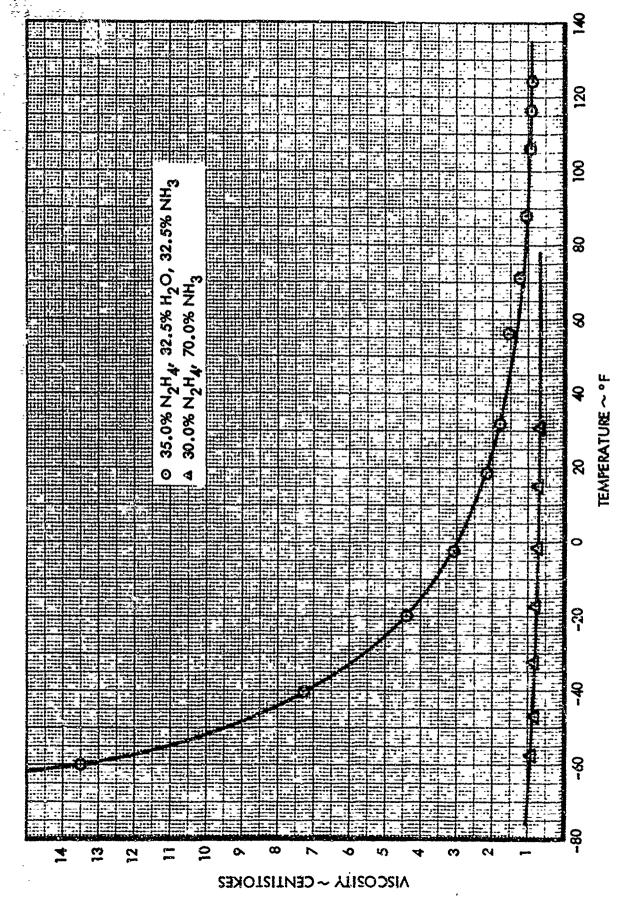


VISCOSITY CENTISTOKES

TEMPERATURE "



TEMPERATURE EFFECT ON VISCOSITY
OF HYDRAZINE - AMMONIA - WATER SOLUTIONS

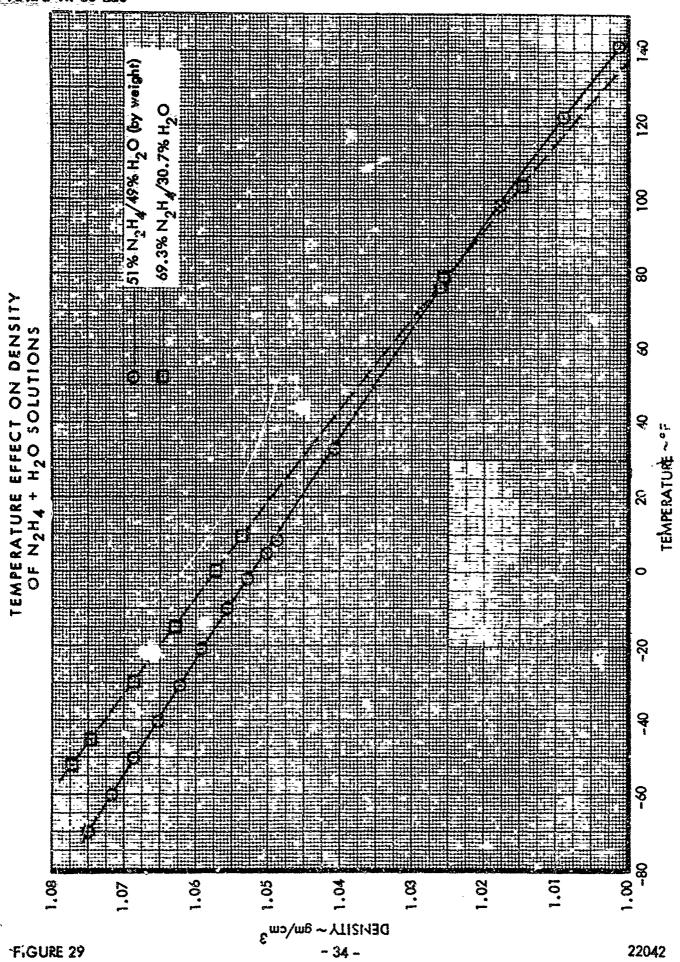


the capillary tubes were closed by pressure stopcocks. This pycnometer was loaded by the same technique used for the rolling-ball viscom. er. The pycnometer was weighed before and after loading and, after thermal eq. librium was established, the liquid level in each arm of the pycnometer was determined by a cathetometer. The pycnometer was calibrated with both mercury and distilled water. This pycnometer was used for the solutions containing 8.75% and 27.5% ammonia. On the latter solution, however, the pressure stopcocks developed leaks at the higher temperatures and the use of this pycnometer was discontinued.

In order to accurately measure the density of the solutions containing 27.5%, 32.5% and 70% ammonia, individual pycnometers were blown from heavy wall glass tubing, and each pycnometer was calibrated with freshly boiled distilled water. The sample was loaded into the pycnometer as before, and after freezing the sample in liquid nitrogen, each arm of the pycnometer was sealed off with a hand torch. As a safety precaution, the pycnometer was then warmed slowly to slightly above room temperature and then cooled to room temperature before weighing. The pycnometers were then immersed in a controlled temperature bath and the liquid level in each arm was determined by means of a cathetometer. It is believed the possible error associated with this method is less than 0.1%. The measured densities are shown in Figures 29 through 33 as a function of temperature.

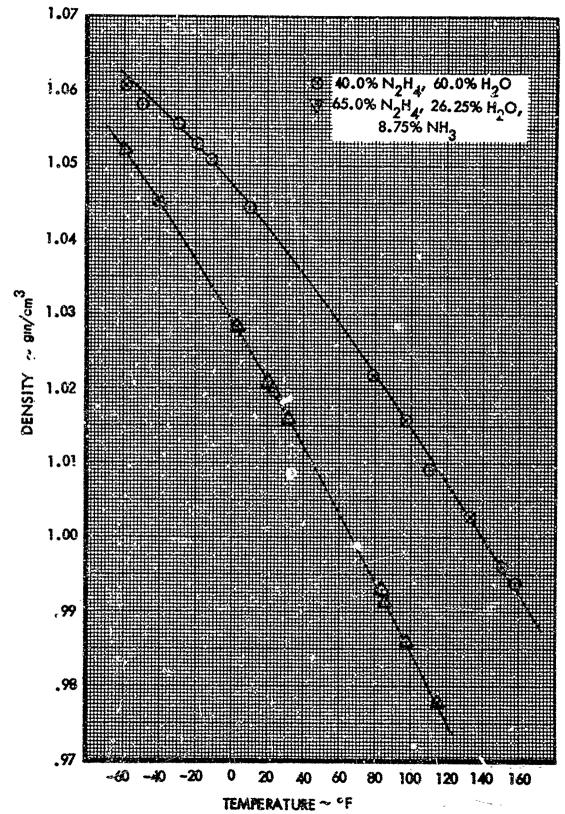
3.4 Summary of Propellant Physical Properties

The compositions and measured physical properties of the seven selected propellants are summarized in Table VIII.



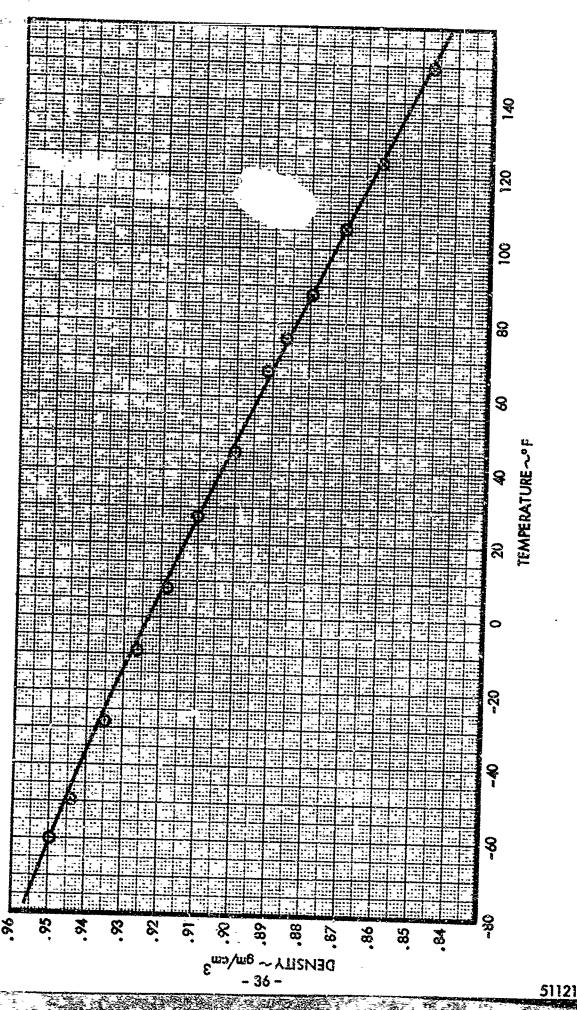
AFRPL-TR-66-226
TEMPERATURE EFFECT ON DENSITY

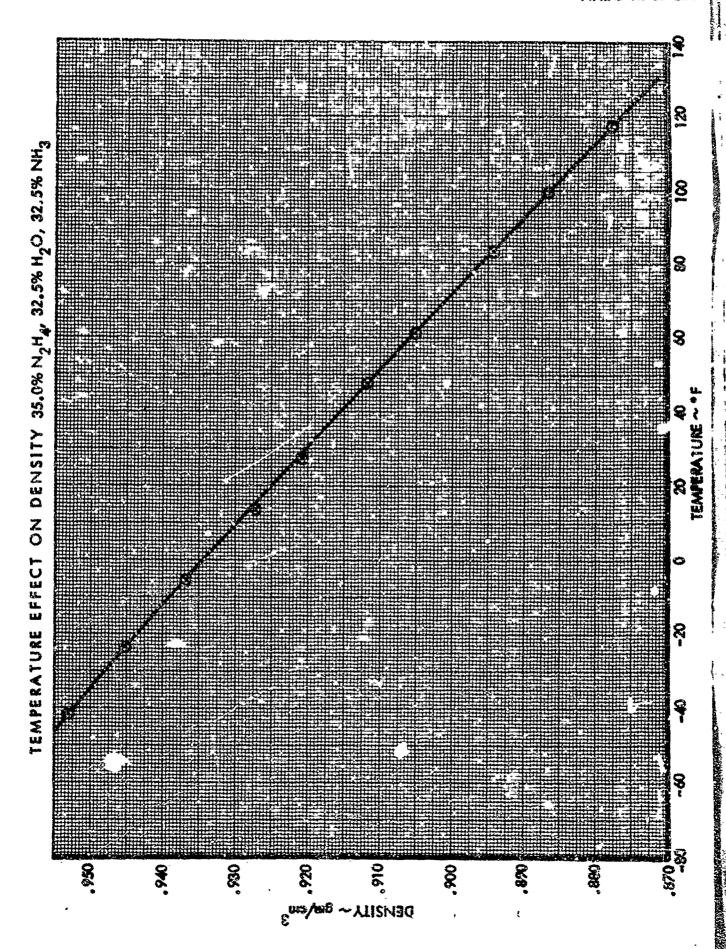
40.0% N2H4, 60% H2O AND 65.0% N2H4, 26.25% H2O, 8.75% NH3



TEMPERATURE EFFECT ON DENSITY 45.0% N₂H₄, 27.5% H₂O, 27.5% NH₃

FIGURE 31





51117

FJG

FIGURE 32

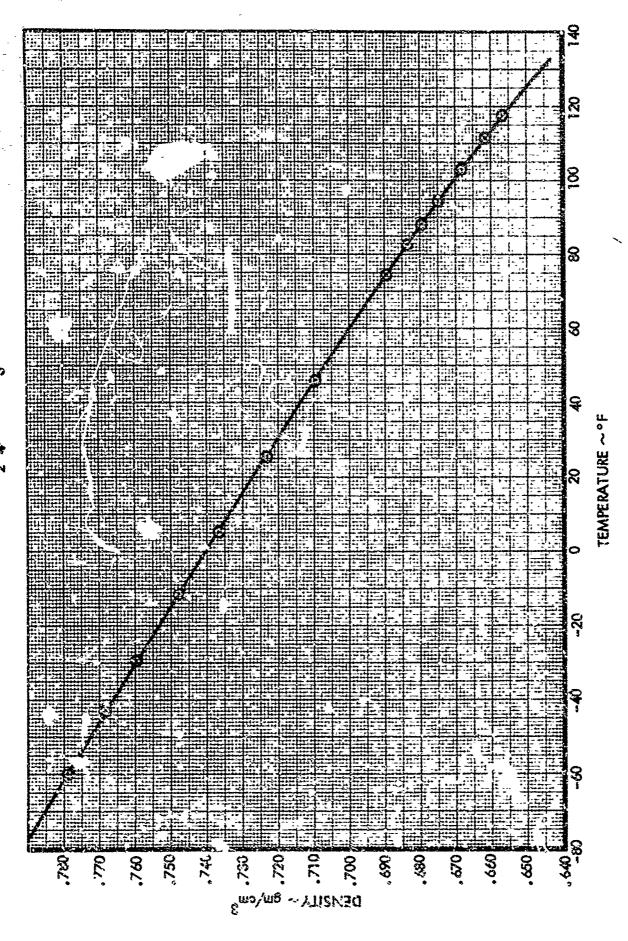


TABLE VIII
PHYSICAL PROPERTIES OF LOW TEMPERATURE GAS GENERATOR PROPELLANTS

×+. % Z±. % Z±4	Wf. % H2O	X. %.	Freezing '~int JF	S	Viscosity Contistokes at °F	sity kes		Density gm/cm ³ ct °F		Vapo	Vapor Pressure psig at °F	ure F
				120	1470	09-	120	+70	09-	120	+70	8
69.3	30.7	į S	-64	1.0	1.8	40.0	1.008	1.029	1.082	8.	.24	° ``
45.0	27.5	27.5) 06-	6.0	1.2	12.0	.883	. 889	.949	84	78	0 ~
o.	32.5	32.5	-104	٥.5	1.2	13.5	928.	0.90	0.960	103	42	°
40.0	0.09	į	-130	0.9	3.	38.3	1.008	1.025	1.064	1.0	.25	0
51.0	49.0	i	08-	0.	8.	48.2	1.016	1.028	1.072	8.	.22	<u>0</u>
30.6	2	70.07	-75	9.0	0.7	9.6	0.654	0.692	0.779	218	16	0 ?
.65.0	26.25	8.75	57	0.	1.5	26.9	.975	666.	1.052	33	7	~ 0

SECTION IV

REACTOR TEST PHASE

4.1 General

Phase III of this program provided for the design and fabrication of a heavyweight gas generator reactor to be used to evaluate the low temperature gas generator propellants whic' were selected during Phases I and II. The propellant evaluation firings were to strong of two, sixty-second steady state firings for each propellant. These tasks were completed as described in detail in later sections of this report.

Due to the completion of another contract (NAS 9-5617) by Rocket Research
Corporation coincident with the start of Phase III of this contract, it was possible to
obtain much more performance data than provided for by the minimum requirements of
the contract. In ddition to the two steady state firings using the Low Temperature Gas
Generator (LTGG) reactor, many firings were made using a 1 lbf thruster unit developed
under the NASA contract. The 1 of thruster was operated for 00 pulses of varying pulse
width and duty cycle on the seven selected propellant mixes, thereby providing much
valuable information on the operating characteristics of the low temperature propellants.

4.2 Gas Generator Design

An assembly drawing of the gas generator is shown in Figure 34. The gas generator consists of three basic parts:

- a. An orificed showerhead injector utilized to distribute the propellant onto the catalyst bed
- b. A cylindrical chamber which contains the catalyst bed
- c. A convergent section which has the capability of replacement of sonic crifices used to control the flow of the gas generator.

The design assumptions and calculations upon which the gas generator is based are described in the ensuing paragraphs.

The gas generator is designed to decompose selected mixtures of hydrazine, water, and/or ammonia as tabulated in Table VIII. The basic design parameters for the engine are: a chamber pressure of 300 psia, a volumetric flow rate of 60 scfm, and an ammonia dissociation with neat hydrazine of 80%. The design of the gas generator is based on

design criteria developed by Rocket Research Corporation under NASA Contracts NAS 7-372 and NAS 9-5617.

Under NASA Contract NAS 7-372, it was shown that a layer of fine mesh catalyst (25 - 30 mesh) approximately 0.2 to 0.3 inches in thickness is required at the top of the catalyst bed to insure smooth and stable operation. The remainder of the catalyst bed is composed of larger size catalyst such that the ratio of chamber diameter to particle size diameter is at least 8:1. A bed loading of 0.0274 was selected for the gas generator. From the definition of bed loading, the chamber diameter is calculated to be 1.50 inches. The catalyst size in the lower portion of the bed is 1/8" x 1/8" pellets (largest size manufactured by Shell Development Company).

The average molecular weight of the gas products from the hydrazine. mixtures is 18.72 if it is assumed no ammonia dissociation occurs as a result of the high water content in the mixtures. Using the perfect gas law, the reactor flow rate is then calculated as:

$$\dot{w} = \frac{P_s \overline{M}}{RT_s} \dot{V}$$

Where:

w = Propellant flow rate, Ibm/sec

R = Universal gas constant

P_c = Standard Atmospheric Pressure, lbf/ft²

T_s = Standard Temperature, °R

M = Average Gas Product molecular weight, lb/lb mole

V = Volumetric flow rate, scfs

Thus:

$$\dot{w} = \frac{(2116)(18.7)(1)}{(1544)(530)} = 0.0483 \text{ lbm/sec}$$

Under NASA Contract NAS 7-372, the following equation was developed to predict ammonia dissociation in the catalyst bed with neat hydrazine and using 1/8" x 1/8" cylindrical catalyst pellets:

$$\ln (1 - X) = -0.472 + 910 \frac{G^{0.71}}{P}$$

Where

X = Fractional ammonia dissociation

G = Bed loading, Ibm/in²-sec

t = Residence time of propellant in catalyst bed, ms

P = Chamber pressure, psia

The residence time is given by:

$$t = \frac{ELMP}{GRT} (1000)$$

where previously undefined parameters are:

E = Fractional catalyst bed porosity

L = Catalyst bed length, in.

Thus, for the design conditions of the gas generator, the catalyst bed length is calculated as:

$$\ln (1 - 0.8) = - \left[0.472 + 910 \frac{(0.0274)^{0.71} (0.340)(13.8)(1000)L}{(0.0274)(1544)(12)(2260)} \right]$$

$$1.6094 = 0.472 + 0.289L$$

L = 3.94 inches

A catalyst bed of 4.0 inches was used for the design. It should be noted that the gas generator was designed for 80% ammonia dissociation with neat hydrazine and that when operated with the propellant mixtures, the ammonia dissociation will be less because of the flame temperature reduction from diluent addition.

The injector design is based on criteria developed under NASA Contract NAS 7-372. This criteria for a showerhead injector is:

- a. Use a pressure drop across the injector of 15% of steady state chamber pressure
- b. Use an orifice density of 6 orifices per square inch of catalyst bed cross-sectional area.

Thus the injector utilizes 12 orifices with a pressure drop of 45 psid. Table IX summarizes the gas generator design parameters.

4.3 Reactor Testing

Schematic drawings of the LTGG and the 1 lbf thruster are shown in Figures 35 and 36. The test set-up is illustrated schematically in Figure 37. The various instrumentation parameters are listed in Table X. A typical run sequence involved the following steps.

- a. Propellant was loaded into the precleaned and nitrogen dried run tank through a 10 micron filter.
- b. Flowmeters were calibrated.
- c. Two 60-second steady state firings were carried out with the LTGG at a nominal chamber pressure of 300 psia. An exhaust gas sample was taken during each firing in a pra-evacuated sample bottle. The gas sample flow was initiated approximately 40 seconds after ignition and sampling was continued until a sample pressure of approximately 50 psig was obtained.
- d. After the second LTGG firing on each fuel mix, the gas generator was removed from the system and even purged at 250°F for 15 minutes with dry, filtered (10 micron) gaseous nitrogen.
- e. A single 30-second steady state firing of the 1 lbf thruster was conducted at a nominal chamber pressure of 70 psia followed as soon as possible by the pulse mode firing sequence listed in Table XI.
- f. Upon completion of the firing sequence, the 1 lbf thruster was removed from the system and oven purged as described in Step d.

4.3.1 Propellant Preparation

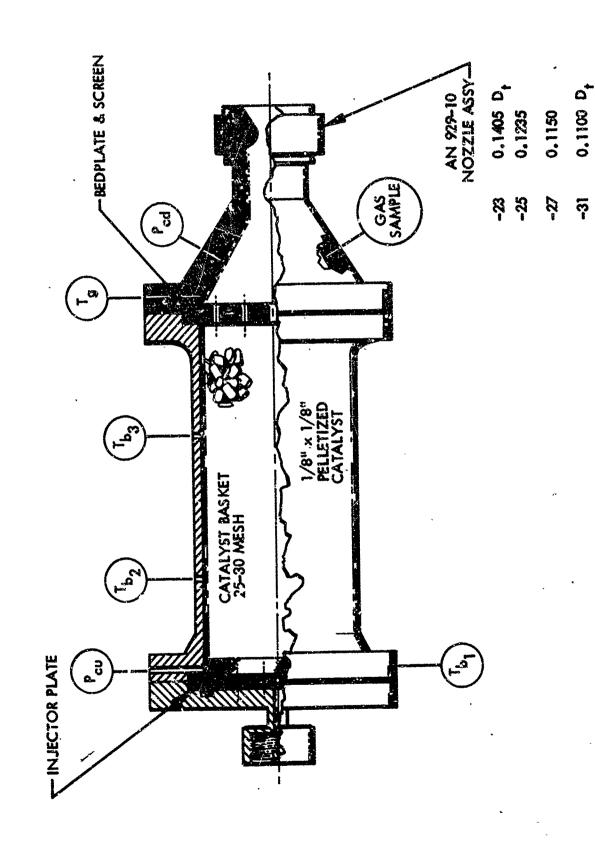
The propellant was prepared by weighing the required amount of water in a polyethylene carboy and adding anhydrous hydrazine by pressurizing the storage

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TABLE IX

GAS GENERATOR DESIGN PARAMETERS

Chamber Pressure 300 psia Volumetric Flow Rate 60 scfm Propellant Flow Rate 0.0483 lbm/sec Predicted Ammonia Dissociation (Neat Hydrazine) 80% Catalyst Bed Diameter 1.5 inches 4.0 inches **Catalyst Bed Length** 0.3 inches 25-30 Mesh Catalyst Size 3.7 inches $1/8" \times 1/8"$ pellets Injector Type Showerhead Number of Orifices 12 Injector Pressure Drop 45 Fsid Catalyst Bed Pressure Drop ~ 4 psid



LTGG REACTOR INSTRUMENTATION

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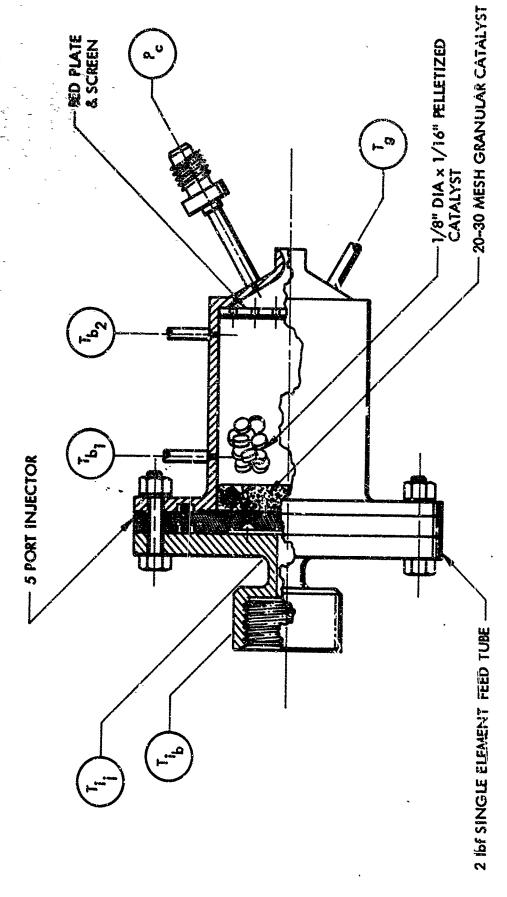
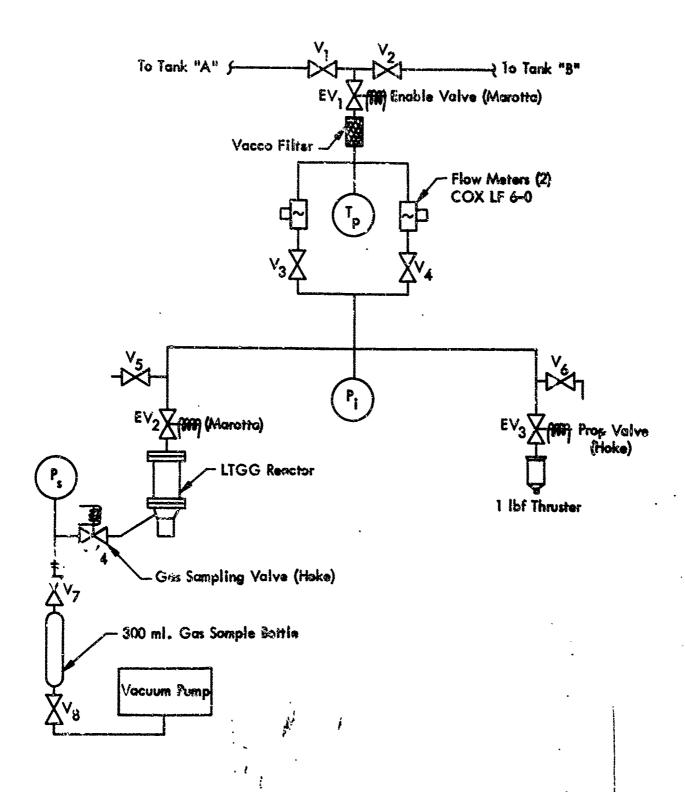


FIGURE 36

- 48 -

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REACTOR TEST SCHEMATIC



51122

- 40 -

FIGURE 37

TABLE X INSTRUMENTATION PARAMETERS

Parameter Symbol	Parameter Identification
P ₁ System	Injactor Pressura
To System	Propellant Temperature
P (LTOG)	Gas Sample Pressure
P _{ed} (LTGG)	Chamber Pressure (Downstream)
P _{cu} (LTGG)	Chamber P. Suure (Upstream)
T _{b1} (LTGG)	Bed Temperature (Top)
T _{b2} (L1GG)	Bed Temperature (MId)
T ₆₃ (LTGG)	Bed Temperature (Bottom)
T _q (LTGG)	Gas Temperature
Wp1 (LTGG)	Propellant Flow Rata
W ₉₂ (LTGG)	Propellant flow Rate
Vpi (LTGG)	Propellant Valve Position
Pa (1 lbf)	Chamber Pressure
T _a (1 lbf)	Gas Temperature
T _{IB} (1 161)	Injector Dass Temperature
T _{lo} (1 lbf)	injector Temperature
T _{b1} (1 16f)	Bed Temperature (Top)
7 _{b2} (1 lbf)	Bed Temperature (Bottom)
= W _{p1} (1 lbf)	Propellent Flow Rute
V _{p2} (1 lbf)	Prepallant Valva Poilton
lu. av	

TABLE XI
PULSE MODE FIRING SEQUENCE

No. of Pulsas	On Time (seconds)	Off Time (seconds)	Duly Cycle %
50	0.25	0.25	50
50	0.25	0,25	10
50	1.0	1.0	30
50	1,0	ዕ . የ	10
25	3,6	3.0	50
25	3.0	27.0	10
20	7.0	7.0	50
20	7,0	0.66	10

container with nitrogen and forcing the hydrazine through a transfer tube into the mixing container until the proper total weight was obtained. The hydrazine was analyzed for water content and the results of the analysis were used in calculating the required weight of hydrazine and water.

The propellants containing ammonia were prepared in the same way to obtain the required hydrezine-water solution. The ammonia (liquid) was then added through a "flexible" line until the required total weight was obtained plus a slight excess. The line was then removed and the excess ammonia was bled off slowly until the required total weight was obtained. Any possible error in weighing due to the influence of the line was therefore eliminated.

4.3.2 Flowmeter Calibration

Calibration of the turbine flowmeters was attempted by flowing the propellant from one presurized tank to another with both tanks presurized to above the vapor pressure of the propellant. A nonstant flow rate could not be maintained by this method, however, and the accuracy of the resultant data was questionable. The propellant flow rate was therefore estimated by utilizing a universal calibration arive which can be readily developed by water calibration. The accuracy of this approach is estimated to be within \$2% and in all probability is better than this value due to the small changes in viscosity of the NH3 mixes as compared to the viscosity of water.

A.4 Results of Test Firings

The results of the LTGG evaluation tests are shown in Table XII, while the results of the 1 lbf thruster test firings are summarized in Table XIII. The theoretical range listed is based on temperatures possible for different percentages of ammonia dissociation. For the LTGG reactor, agreement among the three catalyst bed temperatures and exhaust temperature was reasonably good with the exception of the mixture containing 60% water. In this case, the exhaust gas temperature was from 60 to 160 degrees lower than the three bed temperatures and the theoretical temperature.

The chamber pressure of the LTGG reactor was within 7 paid of the nominal 300 paid except for the two mixtures containing less than 40% hydra time. These mixtures operated

^{*} Plot of cycles per gollon vs. cycles per second/viscosity.

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poorly and had low and decaying chamber pressures. The five mixtures which sustained reactor operation were very smooth running with ahamber pressure roughness factors of within \pm 1% on both resistors except for the mixture containing 65% N_2H_4 . The operation of this mixture was somewhat rougher in the pulse mode tests.

The calculated c* values were in the range of 100 to 106.3% of theoretizal for the LTOG reactor. The fact that the temperatures recorded were near the theoretical upper limit, and the small nozzle prifices used do not make the high c* values too surprising.

The calculated c* values are shown in Figure 30 as a function of weight percent hydrazine. The theoretical curves for hydrazine-ammonia solutions and hydrazine-water solutions are also illustrated for comparison purposes. It is believed that the dip in the theoretical curve for hydrazine-water solutions is a result of the theoretical andensation of water in the chamber with a resultant decrease in the amount of working gas.

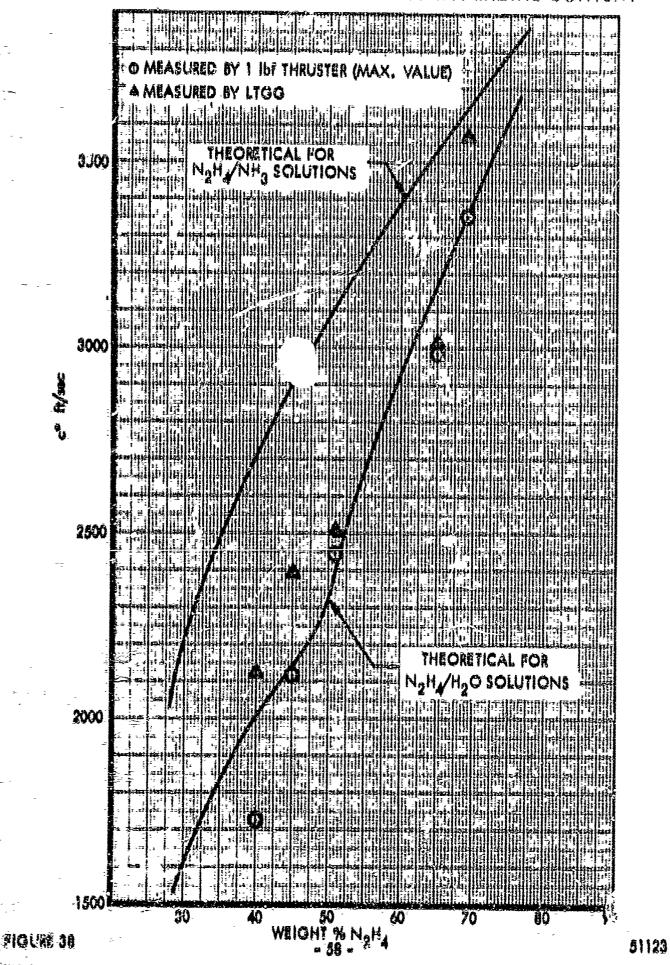
The a* values obtained during pulse mode operation of the 1 lbf thruster are shown in Figure 39 as a function of pulse width and duty eyele. It may be observed that for pulse widths as short as 1 second, the performance approximately equals the steady state values but falls off rapidly for shorter pulses.

The 49% hydrazine-60% water mixture operated smoothly in both steady state and pulse mode operation although very long response times were observed. The propellants containing 30% and 35% hydrazine would not sustain pulse mode operation although the 35% hydrazine mixture could be forced to operate for several pulses by reheating the seach tor externally to ambient temperature.

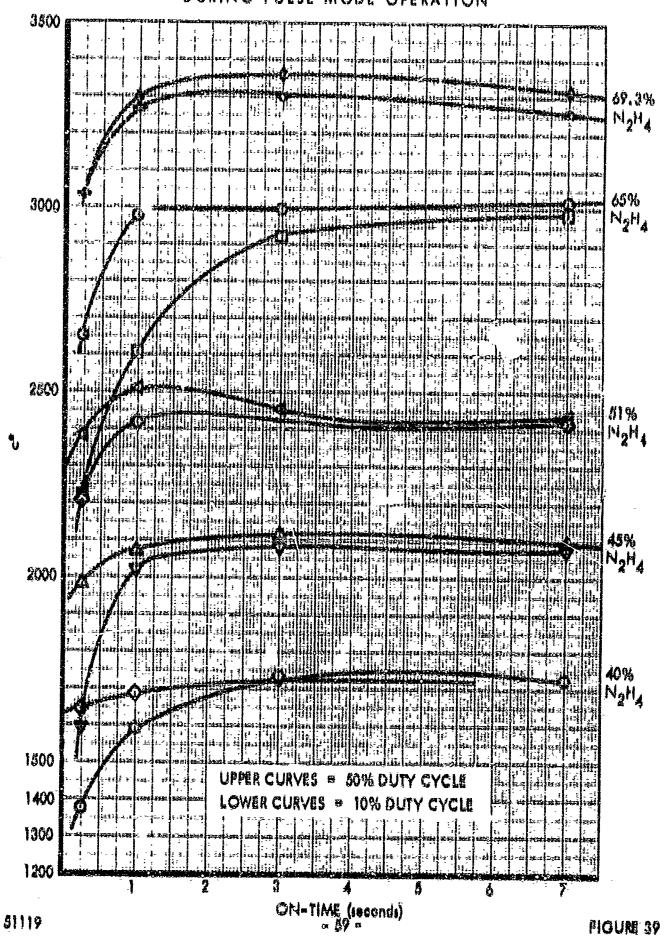
Exhaust gas samples were collected near the end of each 60-seachd firing of the propellant mixtures. The samples were cullected in Hoke stainless steel cylinders of 300 mi capacity which were connected to the reactor (near the nozzle) through a solemold valve and stainless steel tubing. A pressure transducer was also in the system between the valve and the cylinder to manifer the pressure in the sample cylinder. Collection of the gas sample was initiated approximately 40 seagness after ignition and continued until a sample pressure of approximately 50 psig was obtained. The chemical analysis, performed by an independent laboratory, involved heating the sample cylinder to ensure complete vapariation of the sample which was then injected into a gas chromategraph through a heated valve. The results of the initial analyses were not satisfactory, and considerable effort was expended in checking both the analysis and the sampling procedures.

AFRPL-TR-66-226

PERFORMANCE AS A FUNCTION OF HYDRAZINE CONTENT



CHARACTERISTIC EXHAUST VELOCITY DURING PULSE MODE OPERATION



It was not determined whether the sampling technique or the gas analysis was at will. The results of the analyses which were run are shown in Table XIV. These results, while not quantitatively accurate, show the presence of small amounts of hydrogen in the exemples of the higher temperature propulants. The obsence of hydrogen in the exemples of the lower temperature propulants demonstrates that these propellants are operating with no ammonia discoalation as was expected. Additionally, the recorded temperatures and measured characteristic velocities (c*) also confirm these results.

4.5 Summary and Constutions

The modemical calculations have been performed over a wide range of compositions for the system hydrazine-ammonia-water. Experimental reactor firings have been conducted on seven specific monopropellants containing hydrazine, water and/or ammonia. These firings have proven that stable operation is possible over a wide range of propellant compositions. The propellant decomposition products are alean, noncertaine gases in the temperature range of 362°F to 1,160°F. Lower temperatures have been recorded but are still questionable as to repeatability. Two of the seven monopropellants, both containing less than 40% by weight hydrazine, were shown to provide unsatisfactory operation both in steady state and pulse mode systems. The freezing points of the propellants were determined; and their densities, viscosities, and vapor pressures were measured over a wide temperature range. The ammonia and water diluents are excellent freezing point depressints for hydromaline and the propellants exhibited freezing points in the temperature range from -57°F to -130°F.

TABLE XIV
RESULTS OF EXHAUST GAS ANALYSIS

In	itlal Propullant Somposition (Weight %)			Reported C of Exhau (Welg	lomposition* st Quses hit %)	
N2H4	HzO	NHa	N ₂	Hao	NHg	Ha
69.3	30.7	BIX.	30.7	11.4	49.1	0.8
60,3	30.7	ides	39.5	11,4	49,4	0.7
65.0	26.25	0.75	32,8	6.3	60.7	0.2
65.0	26.25	8.75	32.3	6,6	61.5	0.1
40.0	60.0	₩. #	. 14.5	50.7	34.0	Al≅
40.0	60.0		10.4.	43.9	37.7	ă
\$1.0	49:0	.0a-	56.5	16.5	27.0	The state of the s
51.6	49:0	**	29.1	35.5	35.4	
51.0	49.0	30	12.1	60.0	27.8	3. **
61,0	49.0	 #12 .	13.6	45.1	41.0	建 集

^{*}Results are known to be in error due to sampling and/ar analysis teahniques.
See text.

APPENDIX SUMMARY OF PHASE I THERMOCHEMICAL GALGULATIONS

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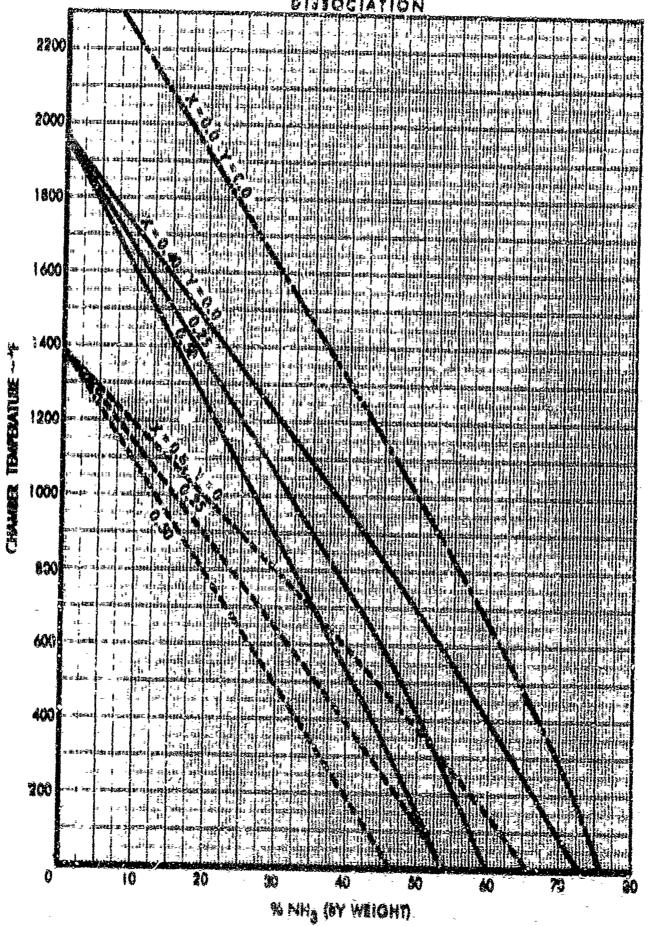
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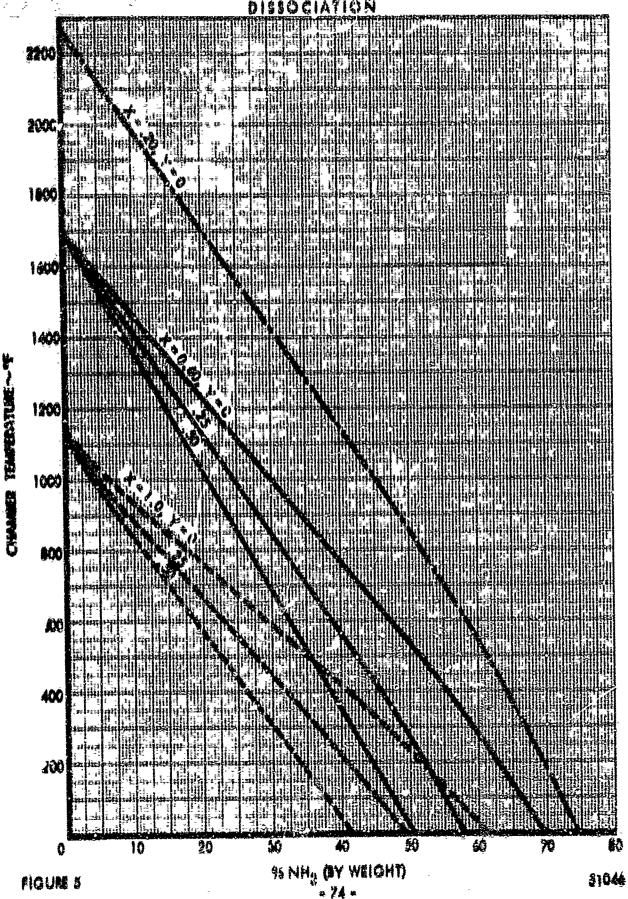
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HYDRAZINE - AMMONIA SYSTEM APPL-TR-66-226
CHAMBER TEMPERATURE VS % NHg FOR VARYING AMMONIA

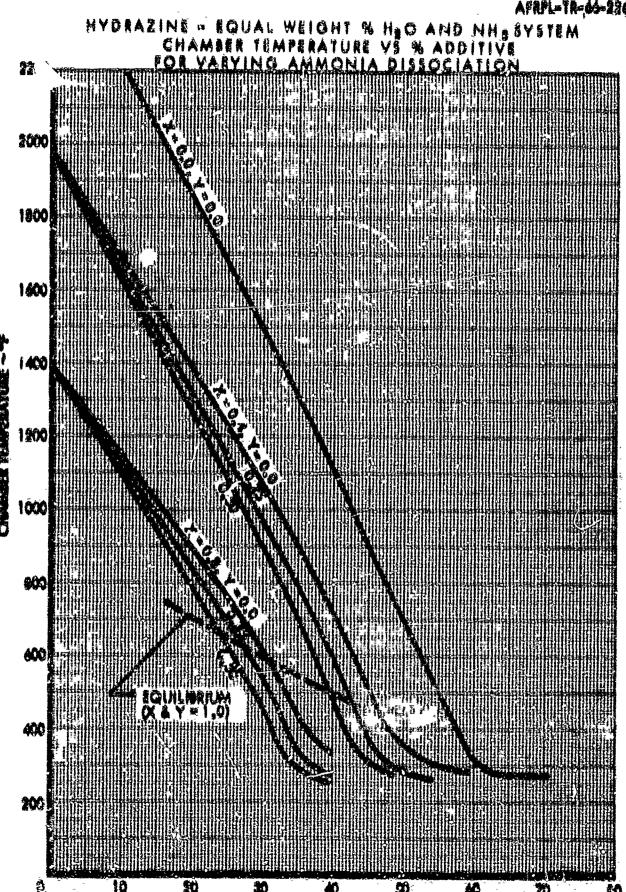


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HYDRAZING - AMMONIA SYSTEM
CHAMBER TEMPERATURE VS % NH3 FOR VARYING AMMONIA
DISSOCIATION

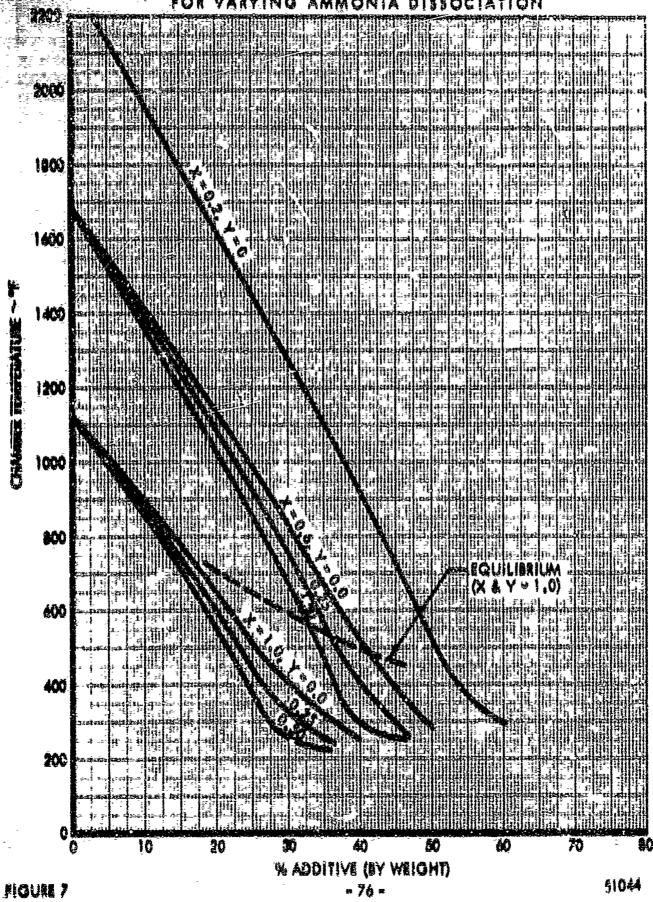


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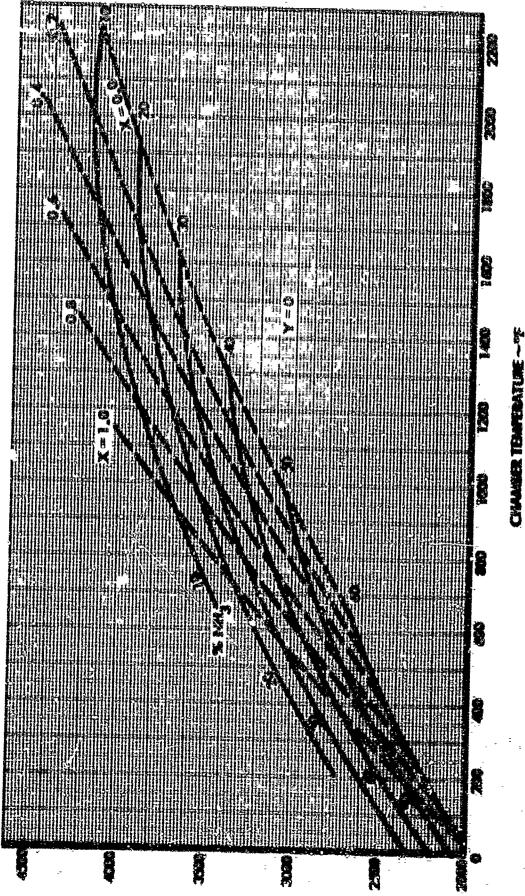


% ADDITUM (BY WEIGHT)

HYDRAZINE-EQUAL WEIGHT & HAD AND NHA SYSTEM CHAMBER TEMPERATURE VS & ADDITIVE FOR VARYING AMMONIA DISSOCIATION



PERFORMANCE OF THE HYDRAZINE - AMMONIA SYSTEM AS A FUNCTION OF TRAFERAL RE AND NH, DISSOCIATION



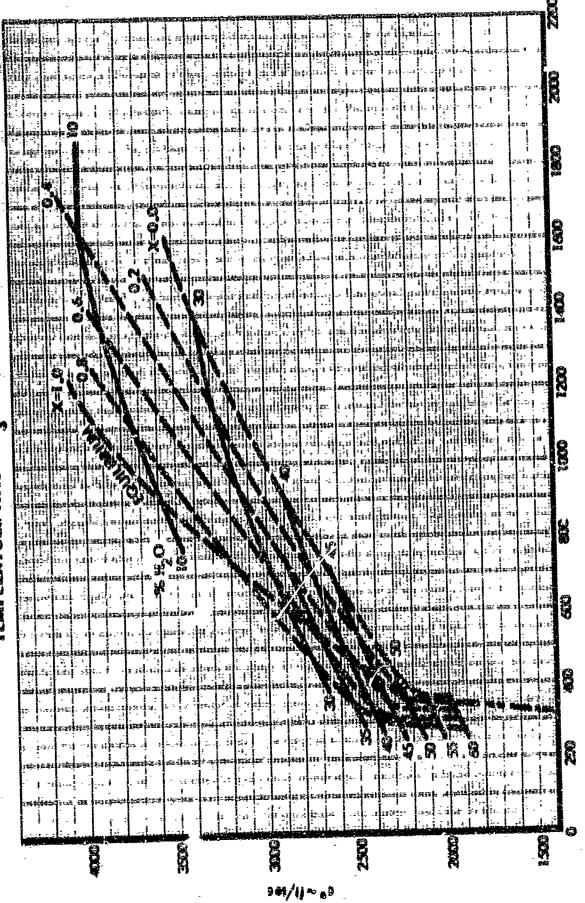
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FIGURE 8

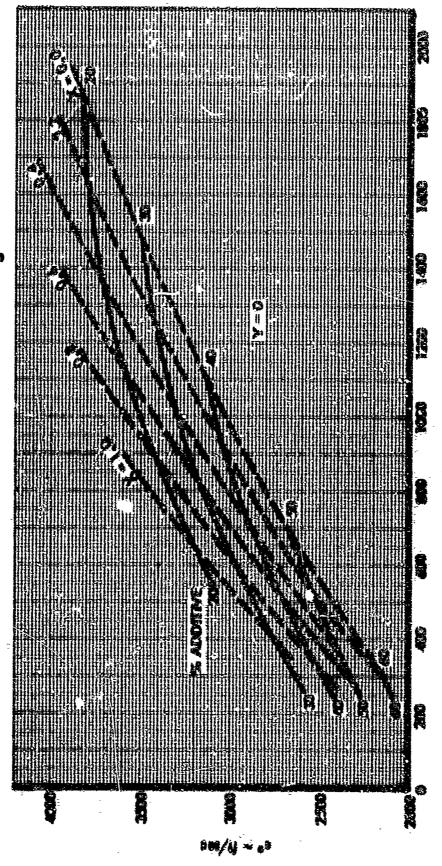
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PERFORMANCE OF THE HYDRAZINE - HAO SYSTEM AS A FUNCTION OF TEMPERATURE AND REJ DISSOCIATION

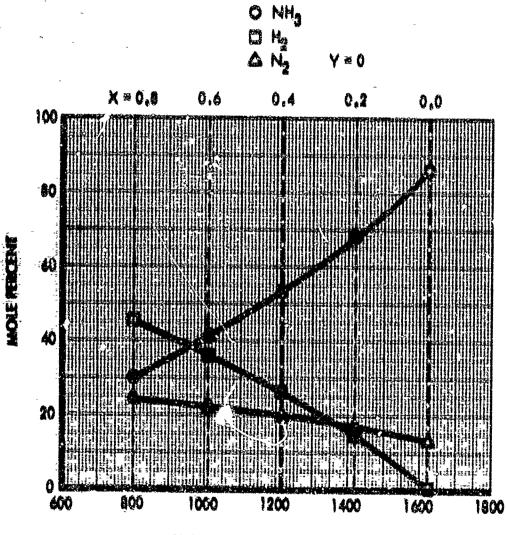


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PERFORMANCE OF THE HYDRAZINE - EQUAL ST. S. M.O. AND NH. SYSTEM AS A FUNCTION OF MEMPERATURE ARE LES DISSOCIATION

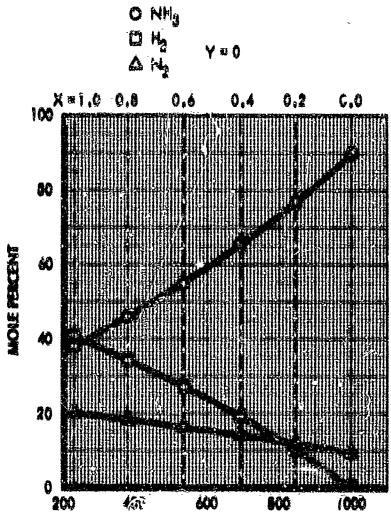


70% HYDRAZINE - 30% NH3 SYSTEM REACTION PRODUCT COMPOSITION



CHAMBER TEMPERATURE ~ *F

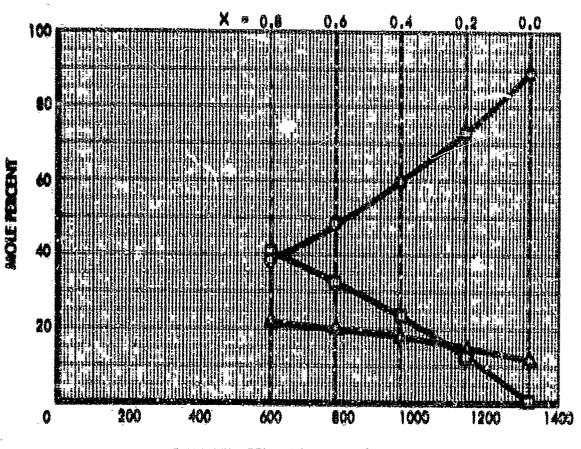
50% HYDRAZINE - 50% NH3 SYSTEM REACTION PRODUCT COMPOSITION



CHAMBER TEMPERATURE ~ "F

60% HYDRAZINE - 40% NH3 SYSTEM

REACTION PRODUCT COMPOSITION

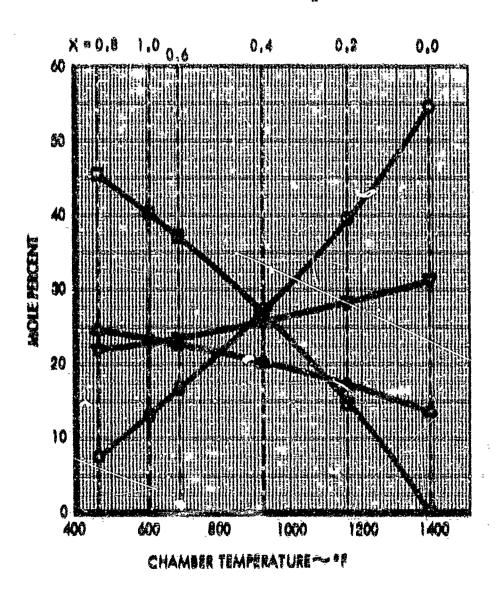


CHAMBER TEMPERATURE ~ *F

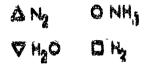
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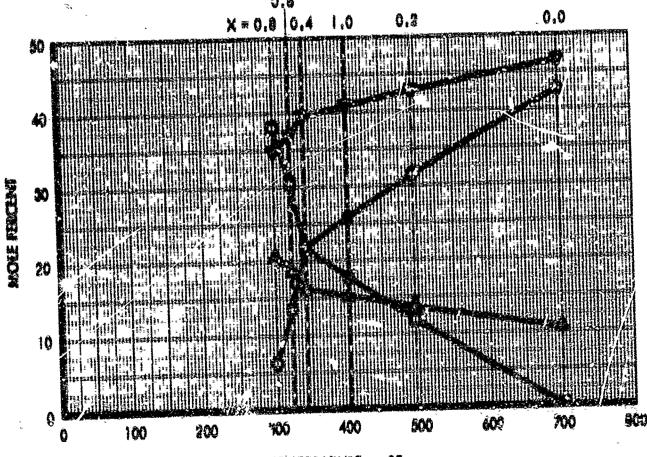
70% HYDRAZINE - 30% H2 O SYSTEM REACTION PRODUCT COMPOSITION

ONH3 & 12 0 H2 V H2O



15% HYDRAZINE - 48% H2O SYSTEM REACTION PRODUCT COMPOSITION





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- 4. Hill, T. G. H., and Sumner, J. P., J. Chemical Society (Landon) Part 1, 1951, pp. 338-640.

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leapable of producing clean, low tempe	iralure gates when passed through a catalylla
idecompatition chamber. During the ex	iurie of the 12×month program, theimachemi=
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columns were determined; and the vap	or prattures, dentities, and viscosities were
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